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A Summary of Current Program 7/1/63

and Preliminary Report of Progress

for 7/1/62 to 6/30/63

EASTERN UTILIZATION RESEARCH AND

DEVELOPMENT DIVISION

of the

AGRICULTURAL RESEARCH SERVICE

UNITED STATES DEPARTMENT OF AGRICULTURE

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This progress report of U.S.D.A. and cooperative research is primarily a tool for use of scientists and administrators in program coordination, development and evaluation; and for use of advisory committees in program review and development of recommendations for future research programs.

The summaries of progress on U.S.D.A. and cooperative research include some tentative results that have not been tested sufficiently to justify general release. Such findings, when adequately confirmed will be released promptly through established channels. Because of this, the report is not intended for publication and should not be referred to in literature citations. Copies are distributed only to members of Department staff, advisory committee members and others having a special interest in the development of public agricultural research programs.

This report also includes a list of publications reporting results of U.S.D.A. and cooperative research issued between July 1, 1962, and June 30, 1963. Current agricultural research findings are also published in the monthly U.S.D.A. publication, Agricultural Research. This progress report was compiled in the Eastern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, Philadelphia 18, Pa.

UNITED STATES DEPARTMENT OF AGRICULTURE
Washington, D. C.
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INTRODUCTION

The mission of the Eastern Utilization Research and Development Division is the development of new and improved products and processes based on designated farm commodities, so as to create new and expanded markets for these commodities. The Division conducts research on dairy products, meat, animal fats, hides, tobacco, maple sirup, honey, and Eastern fruits and vegetables, including potatoes, and new crops.

In carrying out its mission, the Division does research in physical and biological science and engineering throughout the spectrum of basic research, applied research and pilot plant development. Division scientists are aware of the key role that basic research plays in uncovering new information that may be later exploited in applied research and development. Hence, a substantial portion of the Division's effort is in basic research.

The Eastern Division has a total staff of about 440, including some 207 professional research scientists. The Division is organized in 10 laboratories, of which 7 are located at the Eastern Regional Research Laboratory, Wyndmoor, Pa., one is at Beltsville, Md., and two are at Washington, D. C. (of the Washington laboratories, one, the Dairy Products Laboratory, has part of its research program at Beltsville). Two of the Division's laboratories conduct pioneering research, one in animal proteins and the other research on allergens found in certain agricultural products.

In addition to research in the Division's own laboratories, contract and grant research supported by the Division and equivalent to about 13 professional man-years per year is going forward at 16 locations in the U. S. The Division's program is supplemented by a variety of research projects in foreign countries under P.L. 480 grants.

In every phase of their research, Division scientists cooperate with representatives of colleges and universities, State Experiment Stations, research institutes and associations and industrial organizations. Much of the cooperation is informal, but some work is conducted under conditions described in written cooperative agreements and memorandums of understanding. Currently 4 such agreements are in effect.

The farm products with which the Eastern Division deals provide more than half of the nation's cash farm receipts. The major part of U. S. farmland suitable for cultivation is used to provide feed for livestock and dairy cattle; in seven states tobacco provides more cash receipts than any other field crop. Hence it is clear that maintaining and enlarging the markets for these and the other farm products under study at the Eastern Division should be a major national concern.

The opportunities are great. A striking illustration is provided by meat and milk; a one percent increase in meat consumption, which might be

achieved through improved quality, lower processing costs and new product development, would result in an increase in feed use equivalent to 80 million bushels of corn. Similarly, a one percent increase in milk consumption would result in an increase in feed use equivalent to 25 million bushels of corn.

Division scientists have already achieved much both in terms of discoveries now commercialized and discoveries of a fundamental nature that will be exploited in the future. Some recent examples of progress are as follows:

Emulsifying capacity of meat. A simple technique has been devised by EU scientists to rapidly determine the emulsifying capacity of meat from different sources used in sausage formulations. "Emulsifying capacity" is an important property of meat utilized in manufacture of frankfurters, bologna and other emulsion-cured meat products. This development has facilitated the application of automatic data processing to accurate sausage formulation by providing quantitative measurements of the fat-binding potential of the meat. Use of this technique is proving advantageous to sausage manufacturers from the standpoint of cost and overall product quality.

Concentrated apple juice. A commercial installation to produce concentrated apple juice using a process based on EU research is scheduled to begin operation this fall. The factory is now being installed at Cashmere, Washington, by Tree Top, Inc., a growers' cooperative. The new product is a four-fold frozen concentrate with restored aroma. Factory capacity is approximately 3 million bushels of apples annually. It is estimated that apples for the concentrate can yield the grower approximately 50% more than apples used in making the single strength juice currently produced by Tree Top.

Improved evaporated milk. It has been well known that "cooked" flavor which is a deterrent to wider use of evaporated milk as beverage milk can be reduced by using high-temperature-short-time sterilization. However, the improved evaporated milks made by this technique gel rapidly during storage. As a result of EU research it was found that addition of polyphosphates to milk prior to processing markedly reduces the rate of gelation. Practically all U. S. producers of evaporated milk are now testing the commercial applicability of this discovery.

New protein from cow's milk. The existence of a specific "membrane" protein on the surface film of the fat globules in milk has been the subject of speculation and investigation for over 100 years. This protein has now been isolated and characterized by EU scientists. The protein is essentially homogeneous by electrophoretic measurements, and in view of its composition and properties has been classified as a mucoprotein. Antigenic studies showed it to be immunologically distinct from other milk proteins. The mucoprotein proved to be one of the most potent antigens in milk, and hence a potentially significant factor in milk allergy.

Biodegradable detergents from tallow. Current emphasis on biodegradability of detergent materials is causing industry to reconsider tallow as a raw material for manufacture of detergents, wetting agents and other surface active agents. Recent investigations by EU chemists indicate that the α-sulfo acids prepared from tallow are biodegradable as measured by the river dye-away test. From recently completed EU research it is known that α-sulfo acids prepared from tallow are effective lime soap dispersing agents. For this reason, they may be expected to be useful in the form of soap-detergent combinations. This research has also shown that certain esters of the α-sulfo acids are especially effective as wetting agents. Since they compare in effectiveness with the best known commercial wetting agents and are also potentially very cheap to manufacture, commercial interest in them seems assured. In the meantime, manufacture of α-sulfo acids and their esters is being undertaken by several commercial concerns.

It is evident from these examples that the Eastern Division can make highly valuable contributions to agriculture. Indeed, it has been estimated that for utilization research as a whole--adding together the contributions of the Eastern, Northern, Southern and Western Utilization Research and Development Divisions--more than $2.5 billion has been added to the value of products made as a result of the product or process developments of utilization research. This addition represents almost $15 worth of benefits for every dollar spent on utilization research and development in the Department.

AREA NO. 1 AND AREA NO. 2. DAIRY PRODUCTS - CHEMICAL, PHYSICAL AND BACTERIOLOGICAL CHARACTERISTICS; DEVELOPMENT OF NEW AND IMPROVED PRODUCTS AND PROCESSING METHODS

Problem. Dairying is one of the largest segments of American Agriculture: dairy products represent about 13 percent of all farm cash receipts; milk production requires 140 billion feed units annually; milk is a highly nutritious food. It is clear from these facts that research which succeeds in increasing the consumption of milk will have far-ranging effects in raising nutritional levels, in increasing farmers' income, and in increasing consumption of feeds. There is opportunity to increase milk consumption, for per capita consumption is currently at its lowest point in over 30 years at 610 pounds, whole milk equivalent. Current consumption in the U. S. is well below that of several foreign nations, including New Zealand, Canada, Australia, Sweden, Norway and the United Kingdom, all using more than 800 pounds per capita.

Increased consumption can result from improved quality of manufactured dairy products, from cost reductions based on improved processing technology, from the development of new products, or from any combination of these. The development of new and improved processes and products is the objective of utilization research on dairy products.

Both basic and applied research in this field are needed; applied research is the direct antecedent to the development of new products and processes, and basic research provides the information which permits applied research to proceed most effectively.

Increased emphasis on basic research has been advocated by the Dairy Research and Marketing Advisory Committee, and the National Agricultural Research Advisory Committee. Basic research is considered primarily the responsibility of public agencies which disseminate their findings for use by all.

One aspect of the problem posed by dairy products is the great need for fundamental information on the complex biophysical-chemical system which each dairy product is. The development of new products and new processing technology through applied research represents the exploitation of fundamental information. Such exploitation and development cannot continue indefinitely; the supply of fundamental information must be maintained and enlarged, and this is the purpose of basic research. The complexity of milk makes necessary the employment of several scientific disciplines in basic research on this commodity. These disciplines undertake investigations needed to identify and measure the amounts of individual chemical components present; the molecular structure of these components; how the molecules react; and the forces which determine the course of the reactions. These studies should be intensified. Other needed investigations include study of the mechanism of the synthesis of milk; the properties of milk fat; and

the factors responsible for the flavor of dairy products and the changes in flavor which occur during processing and storage.

There is also need for a vigorous and sustained program of applied research which is aimed to increase consumption of dairy products. Such a research program could stimulate consumption by development of products with increased palatability, convenience, or extended shelf life. Another opportunity is the possibility of developing new and improved processing technology which will reduce costs. Because the price elasticity of milk and milk products is greater than that of most food crops, cost reduction is an attractive avenue for increasing consumption.

Still another opportunity is the development of new milk products of low fat content, for example, a low-fat Cheddar cheese. Such a development could alleviate problems posed by current controversy over the effect of animal fats in the diet.

Increased consumption, however achieved, should have a powerful upward effect on feed consumption. It is estimated that milk production requires 140 billion feed units annually (a feed unit is the feed value equivalent to a pound of shelled corn). Hence a 1 percent increase in production would require feed equivalent to 25 million bushels of corn--the production of some 500,000 acres. If the feed were supplied by cropland pasture, more than a million acres would be needed.

It is thus manifest that utilization research leading to product and process development can provide a powerful stimulus to American agriculture.

USDA PROGRAM

The Department has a continuing long-term program involving chemists, biochemists, microbiologists, food technologists, and engineers, engaged in basic research on the composition and properties of milk, and in applied research directed to the development of new and improved dairy products and processing technology.

The Department's research facilities are located in Wyndmoor, Pennsylvania, Washington, D. C. and Beltsville, Maryland. The Federal scientific effort devoted to research in this area totals 73.7 professional man-years. The effort is distributed as follows:

(a) Work on <u>improved concentrated milks</u> involves 11.0 p.m.y. at Washington. Contract research is under way at the University of Illinois, Urbana on possible flavor improvements in concentrated milk; 0.3 p.m.y. is involved. Contract research at Oregon State University, Corvallis deals with methodology for evaluating concentrated milks; 0.4 p.m.y. is involved. Under a research grant to the Ohio State University Research Foundation, Columbus, research is under way (0.8 p.m.y.) on alteration of the inorganic colloidal calcium phosphate complex of milk, with the objective of improving the

stability of evaporated milk. In addition, research sponsored by the Department under a P.L. 480 grant is going forward at (1) Freddo Experiment Station, Milan, Italy on new food products from concentrated milk and fruit juices suited to European tastes; (2) National Institute of Agronomic Research, Paris, France on processing; (3) National Institute for Research in Dairying, Reading, England on the isolation and characterization of selected enzymes of milk to obtain fundamental information useful in improving the quality of dairy products; and (4) Frio Experimental Center, Madrid, Spain on factors responsible for protein destabilization during storage of frozen milk, for use in devising an improved frozen milk concentrate.

(b) Research on dry whole milk and other dried milk products involves 12.0 p.m.y. at Washington and Wyndmoor. The program includes fundamental and applied research directed toward developing technically and economically feasible methods for producing dried whole milk with instant dispersibility and with flavor which will remain stable during six months storage at room temperature and also improved means for drying nonfat milk, whey, ice cream mixes, buttermilk, etc. A research contract with the University of Wisconsin, Madison, provides for a study (10.3 p.m.y.) of the effect of nonfat dry milk on the biochemistry of bread yeast fermentation.

(c) Work on improved butter involves 2.0 p.m.y. at Washington.

(d) Research on improved ripened cheeses involves 3.0 p.m.y at Washington and Beltsville. Contract research on the chemistry of cheddar cheese flavor is being conducted at the Ohio Agricultural Experiment Station, Columbus; 0.4 p.m.y. is involved.

In addition, research sponsored by the Department under P.L. 480 grants is going forward at: (1) Institute of Biochemistry, Turku, Finland on growth-promoting factors for lactic acid bacteria in cheese making; (2) Kaira District Cooperative Milk Producers Union, Ltd., Anand, India on addition of nonfat dry milk solids to buffalo milk in the manufacture of hard cheese; (3) Institute of Dairy Industry, Warsaw, Poland on increasing the vitamin B content of cheese; and (4) National Dairy Research Institute, Karnal, Punjab, India on milk coagulating enzymes of microbial origin, for cheese manufacture. This Institute also has a grant for the isolation and fractionation of components in the proteose-peptone fraction of milk. Additional research under P.L. 480 goes forward at: (5) National Institute of Agronomic Research, Paris, France on the activity of rennin toward individual components of casein; (6) National Institute for Research in Dairying, Reading, England on the application of partition chromatography and allied techniques to the differentiation of microorganisms important in dairy products; and (7) National Institute of Technology, Rio de Janeiro, Brazil on proteolytic enzymes.

(e) Work on flavor components and flavor stability of milk and milk products involves 5.9 p.m.y. at Washington. In addition, research sponsored by

the Department under P.L. 480 grants is going forward at: (1) Biochemical Institute, Helsinki, Finland on the transmission of flavor components and other biologically-active compounds of feed of dairy cattle to the milk and milk products; and (2) National Dairy Research Institute, Karnal, Punjab, India on the effect of the sulfur compounds in milk and milk products on flavor and oxidative stability.

(f) Work on the isolation, structure and properties of milk proteins, and the interactions of milk components involves 20.5 p.m.y. at Wyndmoor, in pioneering studies of milk proteins and investigations of the reactions of proteins and polypeptides with each other and with other milk components. Cooperation is maintained with the Animal Husbandry Research Division, ARS.

(g) Research on the identification and removal of radioactive nuclides from milk involves 8.0 p.m.y. at Beltsville. This is a cooperative program with the Atomic Energy Commission and the U. S. Public Health Service which provides 2.0 p.m.y. additional. The objective is the development of effective ways for removing cationic radionuclides (Sr-90, Cs-137, and Ba-140). A research contract with the Producers Creamery Company, Springfield, Missouri, is concerned with the development of systems for a commercial scale process for removal of radioactive contamination from fluid milk. The contract (7.3 p.m.y.) is supported equally by the Eastern Division and the U. S. Public Health Service.

(h) Research on the control of spoilage organisms involves 1.0 p.m.y. at Washington in basic studies of the formation, germination and heat resistance of bacterial spores.

(i) Work on genetics and milk composition involves 0.5 p.m.y. at Wyndmoor in a study of genetically-caused variation in the composition (and hence the interactions) of selected milk proteins. This work is cooperative with the Animal Husbandry Research Division, ARS. In addition, work sponsored by the Department under a P.L. 480 grant goes forward at the Research Center on Macromolecules, Strasbourg, France, on the structure of nucleic acids.

(j) Pioneering research on the allergens of milk and other agricultural products involves 4.0 p.m.y. at Washington.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Improved concentrated milks.

Recent progress further delineates the range of variations in milk composition and processing conditions that can be used for the preparation of stable HT-ST sterile milk concentrates by use of chemical additives. This work represents the first step towards the production of sterile fluid whole milk concentrates which have satisfactory storage life and will reconstitute to a milk of beverage quality. Experiments indicated that 4:1 sterile skim milk concentrates can be prepared with satisfactory resistance towards gelation,

sediment deposition, lactose crystallization and flavor deterioration. Sterile 2:1 and 3:1 concentrates of "2-10" milks were prepared with storage lives exceeding six months. Sterile high-fat 3:1 concentrates were also successfully prepared. Experiments showed conclusively that under a variety of conditions, substitution of laminar for turbulent flow during the holding period, and of evaporative for forced convective cooling served to prevent undue coagulation during HT-ST processing, and to reduce sediment deposition during storage by 200-300 percent. Hexametaphosphate in concentrates held at 70°F. for two months was largely degraded to pyrophosphate. Trace amounts of pyrophosphate were found in fresh milk and in control concentrates. Such results bear favorably on the question of the suitability of polyphosphates as food additives.

In fundamental research to further elucidate the reasons for gelation and its prevention, it was found that barium and strontium ions effected a marked increase in the storage life of milk concentrates. 0.15% cesium added to milk concentrates brought about a decrease in storage life, magnesium a slight increase. Barium was unique in that its presence did not impair heat stability. Fluoride ions behaved in a manner reminiscent of polyphosphate ions, but were much less effective. Between pH 6.0 and 6.5, the viscosity and storage life of sterilized polyphosphate-containing concentrates were independent of pH. Wide latitude is indicated in variations in composition, and in processing conditions for the preparation of stable HT-ST sterile milk concentrates.

Possible relationship of genetic variants of milk proteins to the processing properties of milk is recognized. However, in the case of two α_s-caseins, designated B/B and C/C, no significant difference in the heat stability and gelation resistance was observed in sterile concentrates containing these variants. Previous work had established the interrelationships of the caseins, phosphate and calcium to gelation and sedimentation in concentrated milks. Further basic research has added important information on the mechanism of this process. The procurement and installation of improved pilot plant equipment during the next report period will extend the study of production variables influencing the physical stability of sterile fluid concentrates containing select chemical additives. The significant factor has been found to be the level of phosphate in the system. The gelation process appears to consist of an interaction between orthophosphate and some internal group of the caseinate particle. As a consequence of this interaction the particles unfold and the unfolded residues are bridged together by calcium into a three dimensional network. The group or groups within the caseinate particle with which phosphate interacts have not been identified. An accurate description of the gelation process should establish guidelines for a rational processing of concentrated milk products so as to avoid gelation.

Under one of the P.L. 480 research grants at the National Institute for Research in Dairying, Reading, England, cow's lipases were emphasized in work done in 1962. Purification of the original extract to a point where

the lipolytic activity had increased 15 times was achieved. Since lipolytic problems in milk may result from high concentration of lipases in only a few animals, many individual milks will be tested.

Under the P.L. 480 research grant at the Freddo Experiment Station, Milan, Italy, new food products, to be prepared by combining concentrated milk and fruit juices, are being investigated. The expected coagulation of casein occurs except with quinces which are high in pectin. Search for preventatives of coagulation has shown that pectin and carboxy methyl cellulose are effective inhibitors while alginates and gelatin are ineffective.

Under one of the P.L. 480 research grants at the National Institute of Agronomic Research, Paris, France, non-protein nitrogen substances which are formed from milk proteins during various industrial treatments of milk are being investigated. Several electrophoretically distinct components were observed in the supernatant after coagulation of kappa-casein with rennin; the rennin-sensitive linkage of kappa-casein is probably an ester between phenylalanine and a hydroxyl group, probably part of an amino alcohol or glucide.

B. Dry whole milk and other dried milk products.

Development of both the vacuum and spray processes for drying whole milk in the form of foams has continued. Several plants have installed the equipment for foam spray drying condensed skim milk and other milk concentrates; one large new plant is designed especially for the foam spray process.

1. Continuous vacuum foam drying. A reproducible set of operating conditions has been developed for "winter milk" which produces a high quality dry whole milk at a rate about 20% higher than the estimated requirement for economical commercial operation. With suitable material thus available, storage tests were begun in which the dry milks are made, handled and packaged with little or no contact with oxygen. For these tests storage is at ordinary refrigerator temperature (40°F.) and at room temperature (73°F.). Residual oxygen content in the cans is less than 0.05% by volume, and oxygen scavengers have not been employed.

Concurrently, statistically designed experiments directed toward optimization of the process are being conducted in the integrated pilot plant. The process must be optimized with respect to 14 currently recognized variables. Thirteen of these variables are controllable. They may be divided into two groups, viz., those characterizing the dryer feed and those representing the drying conditions. Study of the 13 controllable variables for process optimization is simplified by isolating them from the fourteenth (seasonal) variable. However, they can then be studied only during periods of constant foam stability (winter and summer). The seasonal effects must be investigated by comparative studies among the seasons. Since any one seasonal period is relatively short, this investigation must be restricted to that group of variables most closely related to feed characteristics. Moreover,

all pilot plant studies are necessarily coordinated with the laboratory foam stability and composition tests.

From present information there are four distinct seasons with respect to foam behavior in the continuous vacuum dehydrator, i.e., summer, winter and the two transitional periods. The winter season lasts approximately 13 weeks; the spring transition, 8 weeks; summer, 23 weeks; and fall transition 8 weeks. During the winter (low foam stability) season and the summer (high stability) season the foams remain constant in behavior, whereas during the transition periods they change almost daily.

Although a definite statement cannot yet be made, it appears likely that the production rate required for economic feasibility may be attainable even with the more difficult "summer milk" supply. This advance has come about largely through the use of phospholipid (soybean lecithin) as a minor additive to the milk. The most important potential problem associated with phospholipid additive to control foam behavior despite seasonal changes in milk characteristics appears to be that different additive levels may be required at different seasons - unless a level of phospholipid can be found which will mask all seasonal differences. Laboratory research has been expanded to determine whether such a level exists or, if not, what levels of added phospholipid are required throughout the year. This research will be coordinated with studies in the continuous drying plant.

Laboratory observations of milk concentrate foams during the past fall showed the anticipated decrease in foam stability. During this period a rise was noted in the phospholipid content of the milk. Whether there is a cause and effect relationship between foam stability and natural phospholipid level is not certain from available information.

2. Spray foam drying. With nitrogen, air or carbon dioxide injection fresh spray dried whole milk has excellent flavor. The product from nitrogen or air tends to float on water and stirring is required for rapid dispersion; carbon dioxide injection yields a product which reconstitutes more readily and resembles the vacuum shelf dried powder. The nitrogen- and air-prepared samples on storage with 5 percent hydrogen in nitrogen, plus a catalyst in a separate porous container, maintain good flavor for 6 months or more; the powder prepared with carbon dioxide deteriorates more rapidly, presumably because the carbon dioxide is irreversibly absorbed by the powder, resulting in a vacuum within the container thus causing air to leak in during storage.

Foam drying continues to look promising for preparing easily dispersible whole milk powder of beverage quality. Some technical problems remain, but there is confidence that they will be solved. Further improvement in flavor stability during storage is desirable.

3. Flavor. Further investigation of milks made from nonfat solids and butter oil tends to substantiate the hypothesis that chemical changes in the fat phase are largely responsible for stale flavor development in whole milk powders. (This generalization may be limited to powders that have not been

damaged by heat during preparation, that have low residual water content, and are maintained in an oxygen-free atmosphere.) By molecular distillation the off-flavors developed during long storage were removed from nitrogen-packed butter oil. When combined with skim milk the treated butter oil gave a good flavored whole milk. This molecular distillation treatment should be valuable by providing a concentrate of stale flavor components for chemical identification and analysis.

An exhaustive study of ketone formation was conducted. Methyl ketones have been regarded as likely sources of storage off-flavor in whole milk powder. It was found that water is essential for ketones to form in milk fat under the influence of heat. Chemically dried milk fat was very stable to heat in respect to ketone formation. The precursors of methyl ketones have been isolated and identified tentatively as triglycerides that contain a series of beta-keto fatty acids. Water appears to have a catalytic role; it is involved in the hydrolysis of beta-keto acids from the glycerides, a step which is prerequisite to decarboxylation and methyl ketone formation.

A 6-month old foam spray dried whole milk which had been nitrogen-packed and stored at 105° F. was analyzed for carbonyls. No free aldehydes, saturated or unsaturated, could be detected. No dicarbonyls and only a trivial amount of methyl ketones were found. Thus it is possible to virtually eliminate carbonyls as the source of off-flavor in milk powder stored in the absence of oxygen. The relationship to stale flavor of lactones, produced spontaneously from precursors normally present in milk fat, is now being determined.

An oxygen level of essentially zero, which inhibits oxidized flavor development was maintained for 6 months in cans by means of a hydrogen-palladium oxygen scavenging system. This was not achieved with flexible film packages.

Two sets of 50 whole milk powder samples were judged for flavor by a student preference panel at Oregon State University and were scored for type and magnitude of flavor defects by trained panels at Oregon State University and in the Washington and Wyndmoor laboratories engaged in the milk drying research. It has been concluded that the average total score from each of the trained panels will predict satisfactorily the consumer preference; the trained panels' scores for individual flavor defects did not correlate satisfactorily with preference scores.

4. Structure and dispersibility. The film that spreads spontaneously on water from milk powders made with the entire milk fat is being collected in quantities sufficient for a detailed study of its composition. Work on this film material is being intensified since the discovery that powders made from milk containing selected fat fractions do not form surface films which hinder the wetting of the powder mass.

The effect of powder permeability to gases on the flavor stability of spray dried whole milk foams is being investigated. It has been noted that powders produced and packaged under seemingly identical conditions may vary markedly

in flavor stability. The permeability of these powders has also been found to vary. A correlation between these factors is being sought.

Studies of spray dried whole milk foams using gas adsorption techniques indicate that the structure is a gas-in-solid foam characterized by a very low order of surface porosity. This type of structure improves the dispersibility of milk solids but prevents the material from sinking rapidly through an air-water interface. Ideas for producing spray dried particles of the desired type will be tested.

Foam spray dried powders were made from milks containing the individual recently discovered genetic variants of calcium-sensitive casein. No initial differences were noted in the gross characteristics of the milk powders.

5. Other dried milk products. A considerable commercial interest has developed in foam spray drying as a one-step means of making an instant nonfat dried milk. An important aspect of the process is the greatly increased plant capacities made possible because concentrates up 60% solids can be dried. The usual maximum is 45% solids.

The foam spray drying method of making readily dispersible powdered milk has been applied successfully to the preparation of additional dried products such as ice cream mixes, malted milk and cocoa-flavored sweetened skim milk.

It has been discovered that the use of a mixture of compressed nitrogen or air and carbon dioxide results in the production of lower bulk density than when nitrogen or air alone is employed. This means that concentrates which cannot be dried using air or nitrogen injection may be dryable if a mixture of either of these gases and carbon dioxide is used.

C. Improved butter and milk fat products.

Milk fat is a serious surplus problem for the dairy industry; its high price in comparison with competing fats for household spreads is a major reason behind this situation. Research to improve the desirable qualities of butterfat is essential. The Dairy Research and Marketing Advisory Committee has recommended that "basic scientific studies on butterfat should be expanded with the view in mind of defining the various fractions and studying the properties and combinations thereof. It is further suggested that this study include the unique flavor characteristics of butterfat and the potential uses of the various components. Such studies should also include the possible intensification of butter flavor in foods containing butterfat or its fractions."

Development of oxidized and stale flavor in milk fat is a serious storage problem. To date no successful edible antioxidant has been discovered which will prevent oxidized flavor in butter, although antioxidants are widely used in competing household spreads. In concentrated sweetened cream deaeration plus ascorbic acid significantly extended the shelf like of this product at

room temperature. Possible application of this discovery to butter needs further investigation.

Stale flavor in anhydrous milk fat can be removed by steam stripping at temperatures near 200°C.; evidence indicates that precursors of stale flavor are also removed by this treatment, thus suggesting that development of stale flavor during storage can be prevented.

D. Improved ripened cheeses.

That Cheddar cheese made from raw milk has a more intense flavor than cheese made from pasteurized milk is well known. In contract research at Ohio State University chemical differences in the two types of cheese are being investigated. Appreciably greater amounts of free 5-carbon and higher fatty acids have been found in Cheddar cheese made from raw milk. Cheese flavor was not affected by the addition of hypoxanthine, uric acid, acetaldehyde, or cysteine to either raw or pasteurized milk. Improvements were made in analytical techniques for the volatile components of cheese. Through the use of radioactive sulfur it was established that larger amounts of volatile sulfur compounds are present in raw milk cheese.

A low fat cheese of good flavor and body should find a ready market with today's diet conscious consumers. A skim milk cheese with typical Cheddar flavor could not be prepared. Skim milk with enough added fat to give 3 to 4 percent fat in the cheese has been successfully made in the pilot plant into a product with satisfactory body, texture and mild flavor. Some fat is necessary for the Cheddar type flavor; an additive such as a monoglyceride is necessary for the desired body and texture. Additives such as pectin, gum or gelatin improved body, but contributed an undesirable flavor. Proteolytic enzymes, either bacterial or animal in origin, cause bitterness in the aged cheese. Making a good low fat cheese posed new problems both in technology and in microbiology; that is, the standard cheese cultures and making procedures both had to be modified. There is good reason to believe that a satisfactory low fat cheese with Cheddar type properties can be manufactured.

Under a P.L. 480 research grant at the National Dairy Research Institute, Karnal, Punjab, India initial experiments with over 100 isolates of molds, yeast and bacteria to test their potential production of milk coagulating enzymes for use in cheese manufacture, showed that a few molds give positive results when grown on wheat bran extract medium and a few of the spore forming bacteria when grown in milk based media.

Research under a P.L. 480 grant with the Institute of Biochemistry, Turku, Finland showed that the inhibiting effect of serine on growth of *Streptococcus thermophilus* is believed to be due to its stoppage of isoleucine synthesis. Experiments with other microorganisms indicate that serine decreases their production of the enzyme threonine dehydrase.

Under a P.L. 480 research grant at the National Institute of Agronomic Research, Paris, France, a clotting process on treatment of milk with rennin

is being investigated. The reaction which takes place with individual components of casein will be included so that the information can be applied to improving cheese.

The Institute of Dairy Industry, Warsaw, Poland under a P.L. 480 grant has investigated the possible increased synthesis of vitamin B through the use of mutant strains of molds in cheese manufacture. X-rays and gamma rays were used to produce mutations of P. candidum and roqueforti. The promising mutants are being investigated to determine their potential usefulness in cheese manufacture.

The National Institute for Research in Dairying, Reading, England has been studying the application of partition chromatography and allied techniques to the differentiation of microorganisms important in dairy products, under a P.L. 480 grant. By the use of punched cards some correlation was observed between the free amino acids occurring in the cells of cultured bacteria and the quality of the cheese from which the bacteria had been isolated. Some correlation between species and the amino acids present was also observed. Bacteria which contain relatively large quantities of threonine and observable amounts of glutamine were more common in better cheese; also relatively low amounts of aspartic acid and lysine are found more often in these bacteria than in those from the poorer cheese. In relation to the general problem of bacterial flora and cheese quality it is unlikely that a simple survey will do more than suggest a few possible lines of further inquiry.

E. Flavor components and flavor stability.

1. Chemistry. For years researchers in milk flavor problems have felt that carbonyl compounds contribute an important part in undesirable flavors in dairy products. Their formation was explained on the hypothesis that unsaturated fats are oxidized to unstable hydroperoxides which in turn decompose into aldehydes and ketones. Considerable chemical evidence supports this mechanism. Identification in Australia of vinyl n-amyl ketone as a major compound responsible for oxidized flavor in milk fat was an important event in the chemistry of undesirable milk flavors.

The formation of methyl ketones is believed to result from a different mechanism; β-keto glycerides in the presence of moisture can slowly hydrolyze to the free β-keto acids which spontaneously decompose into methyl ketones, more rapidly at higher temperatures. Especially dried milk fat does not produce these compounds even on heating. Qualitatively, saturated ketones do produce some flavor effects in dairy products when present in sufficient amounts; quantitatively, more detailed experiments must be run to decide if these compounds have any influence in dairy products under normal conditions. Lactones in milk were identified several years ago and described as producing a coconut type flavor. Here too, in a quantitative sense, more refined experiments with pure compounds are needed. Much information is available to show that in the complete absence of oxygen stale flavor develops in milk powder. Research in several laboratories indicates that the same or similar compounds are responsible for this stale flavor in both dried whole milk and

evaporated milk; although NFDM also develops a stale flavor on long storage, it is believed that the major source of this flavor is milk fat; skim milk may well contain enough fat to account for this stale flavor. Steam stripping or high vacuum treatment near 200°C. will remove the stale flavor from milk fat; the precursors of this flavor may also be removed.

2. Taste panel. From a contract with Oregon State University, including cooperation of three expert taste panels (Oregon State University, Dairy Products Laboratory in Washington and EU in Wyndmoor) it can be concluded that consumer acceptance of reconstituted dry whole milk can be predicted by well trained "expert" panels. Analysis of the data showed that the average score can be used as a basis for a scoring system which will predict consumer acceptance. However, lack of agreement on description of individual flavor difference of a single sample by different "expert" panels, or even by individual members of the same panel, shows that some problems remain to be solved.

Under a P.L. 480 research grant with the Biochemical Institute, Helsinki, Finland, Professor Virtanen has published about 50 papers based on work done under this agreement. These include important contributions to milk chemistry. Considerable progress has been made toward synthetic diets for cows; if this is achieved many of the problems of milk properties during processing can be attacked more scientifically. Studies of sulfur and selenium metabolism in plants of the onion family are progressing nicely through use of radioactive sulfur and selenium. Enzymatic actions on thioglucosides of the *Cruciferae* family are being investigated; some of these reactions proceed rapidly in the neighborhood of 0°C. The potential goitrogenic effect of milk on humans if the cow's diet contained large amounts of *Brassica* plants has been disproved conclusively with experiments on rats.

F. Isolation, structure and properties of milk proteins and the interactions of milk components.

Three genetic variants of α_s-casein were isolated and partially characterized. Two ribonuclease species in milk were identified by chromatography; one was obtained in pure form and shown to be identical with commercial bovine pancreatic ribonuclease.

A new crystalline protein, named for convenience "lactollin," has been isolated from milk in very small amounts by chromatography on DEAE cellulose. It is a basic protein with a minimum solubility and isoelectric point of about pH 8 and a molecular weight of 43,000. Its amino acid composition is unusual in that it contains no methionine and very little alanine and a large amount of aromatic amino acids.

The red protein of milk, isolated several years ago, has been further characterized by determining its amino acid composition. The distribution of amino acids in this protein shows few unusual features. The basic nature of the protein is, however, reflected in the finding of 25 more cationic than

anionic groups. The red protein was shown to be different from bovine serum transferrins in molecular properties and amino acid composition.

Continued progress is being made in elucidating the amino acid sequence of α-lactalbumin. The amino acid sequence of four pure peptides comprising about one-fourth of the molecule has been completed. With the experience gained in the purification and analysis of these peptides, as well as the improved equipment now available, it is expected that the amino acid sequence of β-lactalbumin will be completed in the not too distant future.

Recent investigations have shown that β-casein occurs in several genetically controlled molecular forms. β-Casein was prepared from the milk of individual cows as well as from pooled milk. Amino acid determinations of these individual β-caseins have not clearly revealed a unique amino acid difference for these forms of β-casein. Another approach to the problem of the genetic forms of β-casein has been investigated. Thus, the large peptides produced by the action of trypsin on β-casein prepared from pooled milk were purified. One of these peptides was found to be present in two homologous forms differing by one histidine residue. The milk from individual cows was found to contain either one or both of these forms. The conclusion from these experiments is that the histidine content of β-casein varies in the genetic forms. Further work is needed, however, to completely clarify the differences in the amino acid composition in the genetically different forms of β-casein.

The antigenic mucoprotein which was isolated from the fat plasma interfacial region of milk has been studied further. Proteolytic digestion of this material, followed by partial purification on Sephadex and ion exchange columns, has given a glycopeptide fraction with a combined hexose, hexosamine and sialic acid content of over 50 percent. The crude digests, resulting from the treatment of interface material with trypsin, chymotrypsin and papain, have strong activity in precipitating antibodies prepared in rabbits against the original mucoprotein. The diffusion technique of Ouchterlony has demonstrated that the digests, like the original mucoprotein, are not immunologically pure. The preparation of a partially purified degradation product of the milk fat/plasma membrane mucoprotein, with immuno- and physico-chemical properties of the interfacial material itself, greatly simplifies a study of changes in these properties during milk processing.

G. Removal of radionuclides from milk.

Research on the removal of fallout from food, especially milk, continues to be a popular subject with public and political overtones. Further important progress has resulted from the joint effort of the Atomic Energy Commission, Public Health Service, and U. S. Department of Agriculture, particularly toward a demonstration of commercial scale use of the process for strontium 90 removal. A contract for this purpose has been negotiated.

The nutritional quality of milk, processed for removal of strontium with the fixed-bed ion exchange method developed at Beltsville, was evaluated by determining growth rates on baby pigs and rats, and by chemical analyses.

The results showed no significant differences between the animals fed ion exchange processed milk and those fed normal milk; there were no marked changes in the vitamin contents of the two milks.

Studies on resin regeneration show that about three fourths of the regenerant (mixed salt solution) can be profitably reused; thus the process requires only about five resin bed volumes of fresh regenerant per 35 volumes of milk. Total cation resin processing costs have been estimated at 0.48 cents and 0.92 cents per quart of milk when technical grade or U.S.P. grade salts are used for resin regeneration, respectively. Investigations were made to avoid acidification and neutralization, which results particularly in increase of the potassium content of the treated milk. Laboratory tests have shown that acidified milk may be successfully neutralized with a combination of calcium hydroxide and sodium hydroxide. A two-step resin treatment procedure which requires no additives for the adjustment of pH was discovered. Milk passed through a sodium charged resin in the "continuous" contactor resulted in about 95 percent removal of strontium. Readjustment in calcium, magnesium, and potassium concentration in the milk was made by passing the treated milk through resin charged with these cations. Flavor of the treated product was acceptable.

In related developments the Civil Defense Division, Department of Defense, is investigating through a contract the feasibility of ion exchange membrane electrodialysis for removing radionuclides from milk. Representatives of industry and government met in October, 1962, to consider the potential usefulness of various methods of removing both cationic and anionic radionuclides from milk. The procedures discussed included ion exchange (column and moving bed systems), membrane electrodialysis and calcium phosphate precipitation.

The principal effort on pilot plant investigations at Beltsville will be to test the recently installed automated equipment which was designed as a complete unit. Conditions for automatically adding acids and neutralizers, and for controlling the sequence of cycles, will be determined. Further evaluation of the milk quality, flavor, removal efficiency, and regeneration requirements will be made. Information from laboratory studies will be used to develop a procedure for the removal of iodine 131 as an integrated part of the decontamination process.

One of the important questions from the public and dairy industry is how soon can milk plants install the equipment. A vital factor in this decision is beyond the responsibility of Utilization Research; government agencies responsible for the safety of food must first decide when treatment is necessary.

H. Control of spoilage.

Bacterial spores, which are responsible for the high temperature treatment necessary to avoid spoilage in canned foods, yield their secrets slowly. Four species of bacterial spores were subjected to extensive electrodialysis.

Gas adsorption and displacement methods were used to determine the surface areas and true densities of three varieties of spores. The data suggest that dipicolinic acid is not held in the intact spore by ionic bonds accessible to water. Studies support the hypothesis that the spore structure can be penetrated by both water and large dissolved molecules. There seems to be little or no apparent correlation between physical surface features and heat resistance. However, large pores seem to exist in the spore wall. Visible verification of spore surface openings and, if possible, their size and number, is being sought with electron microscopy.

This research program on bacterial spores is furnishing basic information on the surface porosity, permeability and adsorbability of spores; and on their content of dipicolinic acid and how it is released on germination. A better understanding of sporulation and germination may lead to a practical means for sterilizing milk and other foods at a much lower temperature than is now possible.

I. Genetics and milk composition.

Recent reported progress in the chemistry and heredity of the lower forms of life suggests that this area may also be fruitful in the search for better milk products. Techniques to produce sometimes useful mutations in microorganisms are already known. To gain control of milk properties through genetics will take a longer time since the generation cycle is in years rather than days or weeks as it is in bacteria.

Investigation of the genetic complexity of the caseins has continued under an examination of caseins from 1,000 cows, including Holstein, Jersey, Guernsey, Brown Swiss, Ayrshire, and Shorthorn breeds. Three types each of α_s- and β-casein are now recognized. These variations are believed to be genetically controlled, as was previously established for the β-lactoglobulins.

Many of the problems in milk processing have never been solved satisfactorily on a year-round basis; processing procedures developed by the trial and error approach have given the dairy industry practical results, but sometimes there is more art than science in the milk plant. Occasionally suspicion has arisen that one or more individual cows may excrete some factor in their milk which has a deleterious effect on processing characteristics, such as heat stability.

Under a P.L. 480 research grant with the Research Center on Macromolecules, Strasbourg, France on kinetics of initial degradation of deoxyribonucleic acid (DNA) by acid deoxyribonuclease it was shown that the reaction occurs according to "single strand" kinetics; this is the first known example of this type of action. Acid deoxyribonuclease from hog spleen was obtained in pure form. Chromatographic studies of DNA have resulted in separation of the denatured substance from the native material.

J. Allergens of milk and other agricultural products.

One human source of strongly reactive antiserum (Acton) and one moderately reactive serum have contributed to development of the program on milk allergens this past year. The high reactivity of the Acton serum with the milk protein α-lactalbumin indicates that either the protein itself is the allergen or the presence of an allergenic impurity in the protein. Collaborative studies with the Department of Immunochemistry at the Walter Reed Army Medical Center have continued on the immunological characterization of the mucoprotein from the fat-plasma interface of cow's milk.

In a collaborative program with Jefferson Medical College investigations on the fundamental basis of milk allergy in infants are being made to ascertain the conditions which lead to the formation of antibodies to cow's milk. Some of the initial problems have been resolved. For example, only a very limited amount of blood can be safely withdrawn from newborn infants. Serum or plasma must be used to assay the milk allergens; plasma is preferable because a greater volume of it can be recovered from blood. Therefore, studies were conducted on the suitability of using plasma in place of serum for the hemagglutination and passive transfer tests which are essential in the tests for milk allergens; it was concluded that heparinized plasma also may be used.

Fractionation and immunochemical characterization of the castor bean allergens have continued. Gel filtration is being used for fractionating the allergenic material; the Ouchterlony double diffusion test is used to determine precipitinogenic capacity of each fraction. Studies of the enzymatic and chemical degradation of the allergen show that it is hydrolyzed to the same extent by the enzymes pepsin and trypsin, but the loss of precipitinogenic activity is greater with trypsin. Hydrolysis with pronase is much more extensive than with pepsin and trypsin, and the loss of precipitinogenic capacity is almost complete. Sodium borohydride, which reductively cleaves proteins at their cystine linkages, completely inactivates the allergen as judged by precipitinogenic capacity.

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AREA NO. 3 - MEAT - PROCESSING AND PRODUCTS

Problem. Livestock production is the greatest single source of farm income: production of meat animals accounted for 30 percent of farm cash receipts in 1961, and the major part of U. S. farm land suitable for cultivation is used to produce feed for livestock. Hence any research which succeeds in stimulating an increase in the consumption of meat and other livestock products can provide a powerful stimulus to U. S. agriculture. For example, a one percent increase in meat consumption would require feed equivalent to about 80 million bushels of corn.

Increases in livestock consumption may be achieved through development of new or improved meat products, or through improved meat processing technology which results in lower costs. In addition, increases in the value of hides, animal fats and renderers' proteins will benefit the livestock industry by providing additional revenues which could permit reduction in meat prices (thus stimulating consumption) or which could flow back through the marketing channels in whole or in part to livestock growers and feed producers. For example, it is estimated that loss of the market for hides would cause an increase of meat prices that would result in a decrease of 2 percent in meat consumption. Such a decrease would eliminate a market for feed equivalent to 160 million bushels of corn. Conversely, an increase in hide values would operate in the opposite direction and would result in greater income to the livestock industry and in increased utilization of feed grains.

The attainment of an increase in livestock consumption requires both applied and basic research. Applied research is the forerunner of commercial practice and is an indispensable element in successful development. But applied research is based on the fundamental knowledge that is acquired through basic research, and represents the exploitation of this fundamental knowledge. The supply of fundamental knowledge must be maintained and expanded, if applied research is to be most effective and fruitful. The need for basic research has been pointed out by the Livestock Research and Marketing Advisory Committee, by the National Agricultural Research Advisory Committee and by other responsible groups.

For reasons outlined above, research which succeeds in increasing meat consumption can have a powerful effect on American agriculture. The potential effect may be assessed from the facts that (1) meat has a high elasticity of demand (a 1 percent drop in retail meat prices will result in a 0.7 percent increase in consumption); (2) the production of 1 pound of livestock requires the equivalent of 7-8 pounds of feed grains; and (3) the U. S. per capita consumption (160 pounds) of meat is no higher than it was 50 years ago and is below that of several other countries, including Australia (234 pounds per capita), New Zealand (222 pounds), Uruguay (234 pounds) and Argentina (166 pounds).

Increases in meat consumption may be achieved by the development of new and improved products that will stimulate demand, or by improvements in processing technology that will lead to reduction in meat prices. The effect of lower prices on meat consumption and on feed utilization is especially striking. Thus, a 1-cent-per-pound reduction in retail meat prices should lead to an increase in consumption of about 1.1 percent. The additional production required to meet this increase will require feed equivalent to about 85 million bushels of corn--the output of about 1.6 million acres.

The attainment of increased consumption will require a vigorous and balanced program of applied and basic research. There is a need for more applied research on processing and preservation, including time-temperature studies of frozen meat, and on new and improved meat products. Of equal or greater importance is the need for more basic research on the physical, chemical and organoleptic characteristics of meat, and the microbiology of meat.

USDA PROGRAM

The Department has a continuing program involving chemists, biochemists, and microbiologists engaged in both basic studies and the application of known principles to the solution of problems in the processing of meat and its products. The Department's research facilities are located at Beltsville, Maryland.

The Federal scientific effort devoted to research in this area totals 19.3 professional man-years. This effort is applied as follows:

(a) Chemical and physical properties of meat proteins involves 4.0 p.m.y. at Beltsville, Md.

(b) Meat flavor and its stability involves 4.0 p.m.y. at Beltsville, Md. In addition research sponsored under a P.L. 480 grant is under way at the Research Institute of Meat Technology, Finland on a study of the influence of microorganisms on flavor development in sausage.

(c) Research on the microbiology of meat and meat products involves 4.0 p.m.y. at Beltsville, Md. A research contract at Iowa State University, Ames, involves 1.6 p.m.y. in a comprehensive study of fungi associated with meat processing and flavor development in order to develop new products of distinctive flavor characteristics.

(d) Study of improved meat products and processing methods involves 4.3 p.m.y. at Beltsville, Md. A research contract at Florida State University, Tallahassee, on a study of the interactions of meat pigments with tissue lipids with the objective of developing methods to control rancidity and pigment fading was terminated. A research contract at Louisiana State University, Baton Rouge, involves 1.4 p.m.y. in investigations on selected characteristics of connective tissue and modification to improve meat tenderness. In addition, research sponsored under P.L. 480 grants is under way at:

(1) Ministry of Agriculture, Cambridge, England on study of the accelerated freeze-drying of meat.

(2) Experiment Station for Food-Preserving Industries, Parma, Italy, on study of the corning of beef.

(3) British Food Manufacturing Industries Research Association Surrey, England, on study of pigment formation and color fixation during the curing of pork.

(4) Low Temperature Research Station, Cambridge, England, on enzymes which attack connective animal tissues, for better understanding of freezer damage in frozen meat.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical and Physical Properties of Meat Proteins

Myosin comprises the larger fraction of fibrillar proteins in the live animal. After slaughter a large portion of the myosin polymerizes with actin to form actomyosin, which makes up a very substantial portion of meat. Myosin also plays an important part in the glycolytic changes which accompany rigor. Its physical-chemical reactions are important in developing a complete understanding of muscle structure and function from the meat standpoint.

Viscosity, sedimentation measurements, and ionization of its phenolic groups indicate that bovine myosin is rod-like in nature, rather than having a random coil. The effects of temperature and change in net charge both support this idea. Of particular interest is the unusual high hydrogen ion transformation, which is indicated by three independent physico-chemical measurements.

Investigations on the binding of electrolytes by meat proteins, and on the retention in meat of various cations show that almost all (90-98%) of the potassium, calcium and magnesium remain in the supernatant fraction on ultracentrifugation of aqueous sodium chloride extracts of meat. In a separate study evidence was found that a large portion of zinc in meat is bound to the salt soluble fraction of proteins.

Because beef fat is less easily used in emulsified meat products than pork fat, work is now in progress on emulsification effects on the properties of these two fats using histochemical procedures. The results throw important new light on the mechanisms of meat curing. The significance of the effect of sodium chloride in releasing calcium, magnesium, and to some extent zinc, lies in the influence of these electrolytes on meat properties.

B. Meat Flavor and its Stability

Both the fat and lean portion of meat contribute to its flavor. Differences

between pork, beef and mutton flavors arise from the respective fat fractions. The dried water extract of the fat-free lean meats on pyrolysis yields identical "aromagrams" in the gas chromatograph. Even whale lean meat gave the same qualitative picture except for trimethylamine, a constituent of many sea foods.

The chemistry of fats seems misleadingly simple on the basis of their glyceride structure alone; glycerides make up about 99% of the total. However, the small remainder, especially the phospholipids, must play a very important part in the storage life of fatty foods. In addition the unsaturated fatty acid components of animal fats play a major role in food deterioration, through the chemical changes, (as distinct from microbiological actions) chiefly multiple and complex reactions with oxygen, that result in formation of minute amounts of carbonyl-containing compounds. Some of these have a drastic effect on flavor when as little as a few parts per million or even parts per billion are present. Also, it seems, a multitude of factors can and does affect the rate, direction and extent of these changes.

The first stages of the fat autoxidation process are believed to lead to hydroperoxide type compounds which are unstable and in turn decompose to chemical substances which undergo still further complex changes which are only partially understood today. The flavor effects of these changes are undesirable and can be retarded in meats but can not be prevented as long as necessary.

The part which aldehydes play in these unwanted oxidative changes in meat is already recognized; additional developments in present research on the further oxidative breakdown of unsaturated aldehydes will increase our fundamental knowledge of the chemical changes which occur in the fats of meat during storage.

The reagents, 2,4-dinitrophenylhydrazine and Girard T, have been used to study carbonyl compounds in foods. The latter gives water-soluble derivatives which are readily convertible to the free carbonyl compounds. The complexity of the mixture of carbonyl compounds in oxidized fat has necessitated development of an involved and time-consuming procedure if each individual compound in each class of carbonyl compound is to be identified and quantitatively determined. Yet the recognized importance of carbonyls, especially unsaturated aldehydes, in obnoxious flavors in oxidized meat products certainly justifies the world-wide attention which these compounds are getting. Unsaturated aldehydes readily decompose in air to rancid-smelling compounds. What these are and how they arise will be extremely interesting future research.

Analysis of the gaseous atmosphere surrounding a food sample in a closed container theoretically will detect the volatile flavor components; however, results will have only qualitative significance, since the concentration of each component in the gaseous phase will be proportional to its vapor pressure. The reported results from lamb fat again show that flavor is very complex, and that even with a tool that may separate all the components into

individual compounds, there is still a tremendous job to identify each one and to eventually resynthesize the original flavor by suitable recombination of the isolated ingredients.

Microbial influences on meat flavor can also be important; however, not only may these sometimes be bad effects, but also enhancement of a desirable natural flavor of meat may be produced. Changes in flavor can be influenced by chemical reactions which occur normally during processing and storage. Cured meats involve the addition of one or more salts which have an effect on growth of microorganisms; obviously if changes in flavor of cured meat are to be produced with microorganisms, strains must be selected which grow in the presence of varying concentrations of salts.

Further progress in this area depends upon additional advances in organic analytical techniques. The fantastic successes with chromatography have made possible the development of present knowledge; the organic chemist must further refine separation techniques, and especially the means of identifying individual compounds in minute amounts. An important complicating factor is the inherent instability of many potent flavor producing substances.

Contract research at Florida State University on the relation of heme pigments to rancidity in cured meats has again demonstrated that a substance which may show antioxidant properties under one set of conditions, may have no protective effect, or even act as a prooxidant, under a different set of conditions; for example tripolyphosphate in cured meats definitely inhibits development of rancidity at one concentration and has no protective action at a higher concentration. Continued research of this type could readily result in practical improvements in storage stability of meat products.

C. Microbiology of Meat and Meat Products

Meat, our most valuable agricultural product, is highly susceptible at all stages of its dressing and processing to microbial deterioration. One estimate of spoilage in the Southeastern states alone is 15 million pounds per year; thus it is readily seen that losses on a nationwide scale are quite serious. Such losses result in increased prices to the consumer which in turn depress consumption and consequently decrease feed grain utilization. Ordinary refrigeration does not control many of those microorganisms which are responsible for much of the spoilage. Changes in fat resulting from both hydrolytic and oxidative enzymes are a major problem. Considerable progress in methodology, both in the microbiological and chemical aspects has been achieved.

In order to carry out comparable assays on the type of lipolytic activity brought about by the enzymes from different microorganisms, a procedure for producing lyophilized preparations has been worked out. These preparations are quite stable and although only partially purified, give reproducible results when assayed at different times. With this method dry enzyme stocks have been prepared from fourteen different microorganisms including bacteria and fungi.

Employing synthetic triglycerides of known composition, the site and amount of activity of each lipase preparation is being determined by gas chromatography. This has involved the preparation, extraction, and gas chromatographic analysis of over 200 samples. When the final calculations are completed the data should afford a good indication of the spectrum of activity that may be expected from microbial lipases. Knowledge of the lipolytic pattern of several microorganisms will permit a more critical appraisal of their possible involvement in the changes in the fats of meats and meat products during storage.

D. Improved Meat Products and Processing Methods

Cured meats, including sausage products, annually produced in this country are in excess of 7 billion pounds, the principle products being ham, bacon, picnics, corned beef, and sausage. This represents more than one third of our pork and an important portion of our beef production, and is a major agriccultural product, with regard to both quantity and economic importance.

In attempting to develop fully the contribution of the meat industry to the agricultural economy, there appears to be a special need for progress in meat curing. Recent changes in the production of cured meats have resulted in reducing curing time and streamlining methods, in an effort to approach a continuous method of manufacture. Results have not been uniformly successful, and it is now doubtful that further developments can be expected from the empirical modification of old processes. Another problem is the fact that the market for cured pork products has fallen below expectations, a particularly serious situation when it is remembered that pork is our principle vehicle for marketing feed grains. The answers to these problems lie in the development of a rational, adequate, scientific basis on which new processes can be developed, one that will provide the rapid continuous processing for which the meat curing industry strives and will yield products of optimum quality and consumer appeal.

The principle objective in streamlined processing is to achieve in hours, or in some cases, minutes, the chemical and physical effects which have previously required up to several hundred times longer. This drastic demand points up the difficulty of adapting old empirical methods to accomplish the desired improvements. A more promising alternative appears to be the development of knowledge of the chemical and physical reactions which should occur in curing and associated processing without time limitations. Following the identification of the reactions, they should be characterized with respect to their kinetics and their variation in different environments. Research of this type should indicate optimum conditions for accelerating the curing processes and provide a rational basis for process modification.

From a contract with Florida State University, Tallahassee, the following findings should be noted: In the study of lipid oxidation in heat sterilized beef it was found that lipid oxidation decreases as the internal temperature of the meat is increased. The production by heat of antioxidant active substances is responsible for the observed oxidative stability. Pigment

destruction was progressive as heat treatment increased but in no case was the pigment destroyed completely. The method for determination of thiobarbituric acid as a measure of oxidative deterioration in cured meats was modified to eliminate the interference of nitrite ion.

The pattern of lipid oxidation was markedly different in cooked and cured meats. The ratio of peroxides to TBA number is 8-10 times as high in cured as in uncured meats. This is attributed to the more rapid decomposition of peroxides by the ferric hemes of cooked meat. Rancid odor showed a highly significant correlation with TBA numbers in both cured and uncured meats. Pigment losses were correlated with both peroxides and TBA numbers. No completely satisfactory antioxidant was found for frozen cured meat. Tripolyphosphate plus ascorbate protected up to 10 to 12 months, but extremely rapid oxidation occurred at that point. BHA prevented rancidity for more than a year, but introduced an undesirable foreign odor after some months in storage. Addition of nitrite without salt gave a stable frozen cured meat without the addition of antioxidants.

Through a P.L. 480 grant to the Experiment Station for Food-Preserving Industries, Parma, Italy, to obtain basic information on changes occurring in beef during the canning process it was found that important adverse nutritional changes occurred during precooking; about one-half of the vitamins and other water soluble components were lost as a result of both heat and leaching effects. Storage at 37°C. for one year produced serious deterioration in edible properties; all samples had a disagreeable taste. At 18°C. the samples were still acceptable after one year.

Through a P.L. 480 grant to the Low Temperature Research Station, Cambridge, England, one good enzyme preparation was discovered that will hydrolyze the connective tissue elastin at meat storage temperatures. Its production, isolation, purification and properties were studied. Possible use of elastolytic enzymes in tenderizing meat will be investigated.

Research under a P.L. 480 grant to the Ministry of Agriculture at the Low Temperature Research Station, Cambridge, England, on the biochemical and physical changes which occur in meat fibers on dehydration showed that beef muscles, whose pH had been raised from 5.5 to 6.5 by injection of adrenaline previous to slaughter, on dehydration by the "accelerated freeze drying" process, are far superior on reconstitution with water, to meat not similarly treated with adrenaline before slaughter.

PUBLICATIONS AND PATENTS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical and Physical Properties of Meat Proteins

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Meat Flavor and its Stability

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Improved Meat Products and Processing Methods

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AREA NO. 4 - ANIMAL FATS AND OILS--INDUSTRIAL UTILIZATION

Problem. The 3.5 billion-pound-per-year output of inedible fats is one of the major products of the livestock industry. It also is one of major concern, because while production of animal fats has doubled in the last 15 years, its principal outlet (in soap) has declined sharply, and is still declining.

The best answer to the question of what to do with huge amounts of fats is to find new uses through utilization research. Already utilization research has played a leading role in finding new uses for over 600 million pounds of animal fats, and thus helped retain markets for fats. There is need, however, for new uses not merely to retain or defend markets, but to expand them, and to upgrade the value of animal fats. The organic chemical industry presents a good opportunity for expanded markets, producing as it does a multitude of products--polymers, plasticizers, insecticides, herbicides, lubricants, paper chemicals--totaling 10 billion pounds. Animal fats possess "built-in" properties which make them potentially useful as raw materials to the chemical industry, but research must be done to realize this potential.

Both basic and applied research are needed; the basic to establish the fundamental facts as to composition, separation of constituents, preparation of chemical derivatives of constituents and determination of their physical and chemical properties; the applied to put these facts to work in developing new industrial products.

An increase of 1 cent per pound in the value of inedible animal fats would provide an additional revenue of $35 million to the livestock industry. This additional revenue will help the industry and growers in the same way as revenue from other animal products and by-products.

The attainment of an increase in the monetary returns from livestock requires both applied and basic research. Applied research is the forerunner of commercial practice and is an indispensable element in successful development. But applied research is based on the foundation of fundamental knowledge that is acquired through basic research, and represents the exploitation of this fundamental knowledge. The supply of fundamental knowledge must be maintained and expanded if applied research is to be most effective and fruitful. The need for basic research has been pointed out by the Commission on Increased Industrial Use of Agricultural Products, by the National Agricultural Research Advisory Committee and by other responsible groups.

USDA PROGRAM

The Department has a broad program of basic and applied research at Wyndmoor, Pennsylvania, and at additional locations where contract and grant research is being carried out involving chemistry and physics, aimed at developing new and improved products from fats for use in industry. Total man-years are 47.9. Of this, 14.2 p.m.y. are devoted to studies on chemical composition and the

physical and chemical properties of animal fats at Wyndmoor. Studies involve fatty acid composition of animal fats using the latest techniques in chromatography and other techniques; intra- and intermolecular structure of pure components and derivatives and factors that influence development of off-flavors in fatty foods. A research contract on the chemical and physical characteristics of organic peroxides involving 0.9 p.m.y. is going forward at the University of Pittsburgh, Pittsburgh, Pa. Additional research sponsored by the Department under a P.L. 480 grant is now in progress at the University of Madrid, Spain, on the preparation of cocoa butter substitutes from animal fats.

Research on improved polymers, plastics, resins and lubricants involving 15.7 p.m.y. at Wyndmoor is conducted on the preparation of new products from fats through vinyl polymerization, condensation polymerization and synthesis of organic compounds for use as plastics, plasticizers and lubricants. A research contract with the University of Arizona at Tucson involving 0.3 p.m.y. deals with copolymerization of products derived from animal fats to synthesize plastics and other polymeric materials. Additional research sponsored by the Department utilizing P.L. 480 funds concerns preparation of hydroxylated derivatives of animal fats for use in industrial products such as plastics and lubricants and is under way at the University of Aix-Marseille at Marseille, France. 6.2 p.m.y. are being devoted at the Laboratory located in Wyndmoor to research on development of improved synthetic detergents based on animal fats, which includes preparation of α-sulfo fatty acids for use in synthetic detergents, and study of soap-detergent combinations for use as bar detergents. 10.1 p.m.y. are being devoted at Wyndmoor to exploratory investigations of new chemical derivatives of animal fats for use as chemical intermediates for industry. A contract with the University of Pennsylvania, Philadelphia, Pa., involving 0.5 p.m.y. provides for determining dielectric properties of long-chain fatty acid compounds and heats of combustion of certain fat compounds.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties: Autoxidation

1. Fractionation and analysis of lipids. Knowledge of the composition of animal fats is of primary importance to a better understanding of their behavior and to improved utilization in both food and non-food products. The development of newer analytical techniques, such as chromatography, has made it possible to separate many of the components of fats that was not possible by the older chemical methods. In carrying out this research a constant effort has been made to improve analytical techniques with particular reference to separation and analysis of fatty constituents.

Studies on the composition and fractionation of fats are being carried out utilizing gas-liquid and thin-layer chromatography and differential thermal analysis. A detailed study of the glycerides as well as the fatty acid composition of lard has been undertaken. Low-temperature crystallization has been employed to resolve the original complex mixture in a number of fractions

so that each fraction will contain only a small number of the principal glycerides. Each of these fractions is then subjected to further analysis to determine individual components utilizing gas-liquid and thin-layer chromatography, X-ray diffraction and infrared spectroscopy. Information obtained from these analytical studies of lard fractions should be of considerable importance in the determination of the complete glyceride composition of lard. They should also result in separation of highly purified natural triglycerides from lard in quantities sufficient to study their structure and physical properties. Thin-layer and gas-liquid chromatography are proving to be increasingly valuable as tools in the study and analysis of natural fats and oils, oxidation products, and products formed during the course of chemical reactions. The findings that epoxy oleic acid is not decomposed or altered during gas-liquid chromatography on special columns should prove of value in furnishing a faster and easier method for analysis of oils containing this and other oxygenated compounds, including commercially epoxidized oils and autoxidized fats.

2. Composition changes of animal fats in foods. Previous studies of fat oxidation have shown that the system is complex and that numerous factors are involved. Further progress has been made in elucidating the role of certain of these factors. Whereas histidine and ferric ions each markedly catalyze the oxidation of emulsified fat, together they exert an effect some six-fold the sum of their individual effects. It was found further that the oxygen absorbed in iron catalysis is all accounted for in the peroxide formed, whereas with histidine only part of the absorbed oxygen is accounted for in the peroxide formed. The type of emulsifier used is also shown to markedly affect the rate of autoxidation. Arginine and histamine combined with ferric ions have little pro-oxidant effect at pH 6.5 whereas histidine is more effective, but at a higher pH (7.5 - 8.0).

The finding that histidine-metal complexes will catalyze the autoxidation of mono and polyolefines is of practical value in that it indicates the complex may be used generally for olefine oxidation. The two-fold action of iron and histidine may make it possible to vary the catalyst concentration to promote oxidation and at the same time keep peroxide formation to a minimum, or to build up the peroxide concentration and keep down secondary oxidation products. The peroxides can be reduced readily to hydroxyl compounds for possible use in industrial applications.

Studies on the action of peptides of histidine in catalyzing autoxidation of emulsified methyl linoleate with ferric ions show a strong pro-oxidative interaction, but not to the same degree as histidine itself. The order of activity of a number of histidine peptides has been determined, the most active being histidyl-histidine. Acetyl-l-histidine has no pro-oxidative action.

The type of basic information being obtained in these investigations should lead to a much better understanding of the mechanisms involved in autoxidation of animal fats in food products, which in turn should permit better control of the process and result in improved utilization of animal fats in

food products.

3. Structural characteristics of animal fat compounds. Basic studies of the structure and properties of animal fat components and derivatives are being conducted through "in-house" and contract research. A study of the structural characteristics of triglycerides is being carried out by Villanova University under a research contract. The research has yielded intensity data with a single crystal of tri-11-bromoundecanoin. The positions of the bromine atoms are being determined, from which will follow the orientation of the fatty chains. Further results have been obtained which throw additional light on the structure of a single triglyceride. If the phase calculations now being made yield the positions of the carbon and oxygen atoms, a signiicant breakthrough will have been achieved.

Previous studies conducted through in-house research on the structure of animal fat compounds have shown that infrared spectroscopy can be used for investigating the structure of diglycerides in dilute solution and for analyzing mixtures of diglycerides, as well as to study the interaction of the OH group with its intermolecular environment. More recent investigations have concerned determination of the infrared spectra of model compounds and interpretation of the data in considerable detail. Quantitative conclusions also have been drawn from an infrared study of the fundamental hydroxyl stretching vibrations of derivatives of fats and related compounds.

The data on fatty compounds being developed under this project are of an extremely fundamental nature and should lead to a much better understanding of the ultimate structure of fat compounds, and should add to the fundamental information on the behavior of fatty compounds. Information on the basic structure of the esters and hydroxyl derivatives is important as a basis for research on industrial utilization of chemical derivatives of fats.

B. Improved Polymers, Plastics, Resins and Lubricants

1. Plastics. Since the plastics and plasticizer fields offer large potential outlets for chemical intermediates derived from animal fats, a wide variety of components and derivatives of animal fats are being investigated for use in these fields.

(1) Fat derivatives for polymer modification. Studies have been carried out on preparation of polyhydroxylated derivatives of fats by acid-catalyzed hydration of internal epoxides. Under certain conditions ether type compounds are formed. Further study of the reaction has shown that epoxidation of methyl linoleate results in formation of two isomers, a solid and a liquid. The liquid isomer has been prepared in 85% purity. Protective coatings have been prepared from resins derived from condensation of formaldehyde with hydroxyphenylstearic acid. Further studies have also been made of the rearrangement of epoxy esters to keto esters. The formation of intermolecular and cyclic ethers from epoxides may provide fatty derivatives of considerable value in industry. Further characterization of the compounds must be carried out before assessment of their value can be made.

(2) Vinyl copolymers from animal fats for use in plastics. Preparation of these compounds is being studied under contract research with the University of Arizona, supported by the four Utilization Divisions. A number of vinyl substituted monomers have been polymerized and the polymers, both homopolymers and copolymers, evaluated. Vinyl tetrachlorostearate capable of homopolymerization has been copolymerized with vinyl chloride. Vinyl dichlorostearate, in a polyvinyl chloride copolymer, has internal plasticizer action, but less than vinyl stearate. Examination of copolymers of vinyl chloride and vinyl esters of long chain fatty acids (C_{16} - C_{22}) shows that long chain co-monomers are more effective internal plasticizers, but with increasing ester content the tensile strength of the polymer decreases. The information acquired on homo- and copolymerization is part of an evaluation of these monomers in the polymer and plasticizer field. The information obtained contributes further to evaluation of these fat-based monomers for use in the plastics industry. Further insight into the possibilities of internal plasticization by means of vinyl substituted fatty monomers will better define their commercial value. Additional information on the present monomers and their products, as well as other fatty monomers, will be sought. The effect on properties exerted by polar substituents such as hydroxyl in the fatty side chain of copolymers will also be examined.

(3) Preparation of organic-inorganic type compounds from fats for use in the plastics field. This work has concerned compounds and their derivatives prepared by addition of dichlorocarbene to unsaturated fat compounds, particularly the esters of unsaturated fatty acids. It has been shown that with purified fatty esters yields of 75 to 88% are obtained. The reaction is being extended to intact naturally occurring fats and oils. Certain chemicals (sodium methoxide and dichloroacetate) form dichlorocyclopropane rings at the unsaturated groups of the fat compounds and transfer the fat to the corresponding ester in 80 - 90% yield. With white grease about 90% of the double bonds are converted to dichlorocyclopropane rings. Extension of this addition to the native fats and oils permits forming dichlorocyclopropane rings in both mono and polyunsaturated fatty acids. Of the resulting compounds, those containing several dichlorocyclopropane rings are soluble in silicone fluids, which suggests applications as plasticizers or lubricant additives. This work has added two new methods to derivative-forming reactions of fat chemistry--the preparation, using dichlorocarbene, of dichlorocyclopropanes, and the reaction of the latter to give β-chloroallylic ethers or alcohols. These reactions and their products, from the standpoint of multiple and unusual functionality, may prove of use in various fields such as polymer, plasticizer and perhaps lubricant.

(4) Preparation and polymerization of polymerizable amides from animal fats. This work has concerned the polymerization characteristics of N-allylamides catalyzed with benzoyl peroxide. The benzoyl peroxide radical reaction on amides was characterized by the occurrence of a number of parallel reactions: (a) formation of benzoic acid and a dimeric compound possessing monomer characteristics; (b) decomposition of peroxide by the amide radical to give aryl substituted monomers; and (c) simultaneous polymerization of these monomers. In the course of the study of the homopolymerization of

N-allylstearamide, it was found that by using a small amount of initiator (starter) hard, high-melting waxy product mixtures were obtained. These products are being evaluated. N-octadecyl acrylamide yields copolymers high in molecular weight, regardless of the amide content, and may be expected to modify commercial polymers without the serious molecular weight reducing limitations shown by N-allylstearamide. Its copolymerization parameters indicate suitability for copolymerization with many commercially available monomers.

2. Lubricants. The field of lubricants and lubricant adjuncts is very active, especially with respect to developing new and improved lubricants to meet the far more exacting requirements of high performance engines of all kinds. This area offers considerable promise for increased uses for inedible animal fats. Previous studies have shown that a large number of compounds prepared from α-sulfo fatty acid esters, aromatic adducts of methyl oleate and others, when tested, were found to have little potential as anti-wear lube adjuncts. Work has continued on preparation and testing of additional compounds. Esters of benzyl, α-toluene thiol, isopropyl benzyl alcohol, anisyl alcohol and benzhydrol with dimer acids have been prepared in nearly quantitative yields by conventional esterification procedures. The dibenzyl esters of hydrogenated dimer acids and sebacic acid were also prepared. Thermal stability and wear tests, important tests in evaluating lubricants, were made on the compounds. The wear test on the esters compared favorably with control runs using dioctyl sebacate and paraffin oil. The benzyl and anisyl esters had wear characteristics far better than either of the standards. With increased knowledge and experience in testing various types of fat derivatives, it should be possible for products to be "tailor-made," which will have value as lubricants or lube additives. So far diesters and compounds containing phosphorus have shown the most promise.

C. Improved Synthetic Detergents

1. Soap-detergent combinations from inedible animal fats. Past research has shown that α-sulfo tallow acids have possible applications in solid detergent compositions and detergent bars. Investigation of liquid detergent formulations and ease of their biodegradation has been initiated. Formulations containing mixtures of esters of α-sulfo acids and sodium oleate were found to be stable and have acceptable detergency in hard water. Biodegradation studies of detergents in river water indicate most fat-based detergents are easy to degrade compared to petroleum-based synthetic detergents, which are "hard" and may create problems in sewage disposal plants and water purification. From our past and present research on synthetic detergents from animal fats, the monoesters of certain type compounds ($RCH(SO_3Na)CO_2R'$, where R' derives from a lower molecular weight alcohol or sodium isethionate) seem to have the best properties and possibilities for detergents, perhaps as soap-syndet or "combo" bars, or in solid or liquid detergent formulations. Our research has shown that many biodegradable detergents from animal fats are sufficiently soluble to be components of liquid "syndets," and studies to determine the most appropriate formulations are in progress. A recent review and forecast of future detergent trends suggests that esters of α-sulfo

tallow acids might become commercial products. Greater knowledge of the relation between structure, properties, and performance, including ease of biodegradation, will facilitate commercial development.

D. New Chemical Derivatives

1. Addition of nucleophiles. The preparation of glycols and m-dioxanes in high yields by addition of formaldehyde to the double bonds of unsaturated fatty acids should provide compounds of wide utility in areas such as polymers, lubricants, plasticizers and functional fluids. However, the reaction yields a complex mixture, and it is essential to develop methods for separating the glycols from the dioxanes and to ascertain the chemical and physical characteristics of the substances before consideration can be given to their utility. While considerable progress has been made in this direction, the problem has not been completely solved. The work under this project was discontinued during the reporting period. The chief accomplishments resulting from the work were: (1) Phenol and substituted phenols were successfully added to the double bonds of oleic acid and oleates; (2) Oleic and other unsaturated fatty acids and esters were carboxylated with carbon monoxide, thereby converting monobasic acids to dibasic acids; (3) A detailed study of the addition of formaldehyde to oleic acid has shown the reaction to be extremely complex, yielding numerous products difficult to separate, with no single one predominating.

2. Phosphorus, sulfur and nitrogen-containing fat derivatives for plastic intermediates. This area of exploratory research concerns the synthesis of phosphorus, sulfur, oxygen and nitrogen-containing compounds from animal fats by various reactions. During the reporting period work has been carried out on preparation of difficult to obtain long-chain fat compounds through decomposition of organic peroxides; free-radical addition of various addenda to unsaturated fatty acids and their esters; addition of thiocyanogen to methyl oleate; diacetyl peroxide reaction with iodine to yield alkyl iodides; and the acylation of amides and imides with isopropenyl esters. New peroxy acids that have been prepared should permit more rapid oxidation of long chain compounds that have hitherto not been oxidizable to products useful in the plastics field. In all cases new procedures are being developed for the introduction of reactive functional groups by efficient means into relatively unreactive molecules. Products are either bifunctional or have reactive handles, which permits their use in numerous important industrial outlets. The development of new reactions of fatty acids is essential if their utilization is to be increased. The preparation of dibasic acids directly from unsaturated acids by free radical addition promises to be a most valuable reaction, but it will require considerable effort to work out the details. Thiocyanogen addition products should be good precursors of thiiranes for which there is active industrial interest.

3. Dielectric properties and structural characteristics of organic peroxides.

Fundamental studies on the crystal structure and basic physical properties of organic peroxides are being carried out through contract research at the

University of Pennsylvania and at the University of Pittsburgh. Organic peroxides derived from animal fats are easily and inexpensively prepared, and they are labile compounds which undergo a wide variety of organic reactions. These studies will provide basic information on the structure of these peroxide derivatives and important thermodynamic data which will aid in the development of reactions for converting peroxides of fats and their derivatives to more useful chemicals. Considerable progress has been made in the studies on determining the dielectric properties of fatty peroxides. Heats of combustion of peroxy acids and t-butyl peresters and the kinetics of decomposition of the t-butyl peresters have been completed and the results published. The heat of fusion of peroxylauric acid has been determined, which completes this study on the heats of combustion of peroxy acids, peroxy esters and related parent compounds. The experimental determination of the heat of fusion of peroxylauric acid aids in eliminating a serious error in calculation of the o-o bond energy and the chelation energy of the intramolecular hydrogen bond. The results obtained in this study are of a fundamental nature, and their evaluation must await completion of the work. Studies on the crystal structure of organic peroxides being conducted at the University of Pittsburgh have been under way a relatively short time and results are only preliminary. A determination of the absolute configuration of organic peroxides is essential, not only in the development of new useful compounds, but also will contribute to our basic knowledge in this field.

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AREA NO. 5 - HIDES, SKINS AND LEATHER - PROCESSING AND PRODUCTS

Problem. To maintain the utilization of animal hides and skins at a profitable level there is need to find new products and processes to provide outlets for about 7 million cattlehides that are now available in excess of domestic needs. The foreign markets that currently absorb these surplus hides are also threatened by the increased hide production and decreased per capita use of leather (the principal outlet for hides) that have dislocated U. S. markets and caused prices to drop so precipitously in the last 10 years. To meet this problem there is need for upgrading the quality of raw hides and skins, for reducing the quality of raw hides and skins, for reducing the costs of producing leather, and for developing products with improved properties. To achieve these objectives research is needed to develop improved curing processes and agents, more effective control measures for (ante mortem) defects such as grubs, brands and parasite damage, and improved methods of take-off. Fundamental research is needed on the composition of hides to provide basic information on the chemical, physical and physical-chemical properties and reactions of collagen and other hide components for use in studies on chemical modification and on the development of new and improved products and processes. Development of new, more rapid and economic processes for curing, handling, unhairing and tanning hides is needed to reduce the cost of producing leather. There is also need for research on the chemical modification of hide proteins to develop leather products with such improved "built-in" properties as increased resistance to wear, scuffing and deterioration from perspiration, enhanced washability, dry-cleanability and improved dyeability.

USDA PROGRAM

The Department is conducting a broad program of basic and applied research on hides, skins and leather at Wyndmoor, Pennsylvania; and at additional locations where contract and grant research is being carried out; which involves chemists, biochemists, microbiologists and leather technologists.

The Federal scientific effort devoted to the overall program totals 22.9 professional man-years, as follows:

(a) Chemical and physical properties and structure of hides and collagen involve 8.7 p.m.y. at Wyndmoor. These investigations encompass exploratory research on cattlehide components and basic research on the chemistry of collagen. One line of investigations is concerned with the isolation and identification of cattlehide components, their chemical and physical properties, and their organization within the hide structure as related to leather properties. The second part of the program deals with the forces that control the stability and reactivity of collagen and the factors responsible for the unique physical properties of leather. Of special interest are the physical-chemical properties of collagen, its soluble components and its reaction products with modifying chemical agents. A contract with the Franklin Institute, Philadelphia, Pennsylvania, utilizing 0.7 p.m.y. provides for the development of a dynamic mechanical tester to be used in studying the

fundamental physical properties of leathers and polymers. The University of Cincinnati, Cincinnati, Ohio, is conducting contract research on the non-collagenous proteins of cattlehides involving 0.4 p.m.y.

In addition, research sponsored by the Department under the P.L. 480 program is in progress at:

(1) University of Turku, Finland, on the fractionation of gelatin and soluble collagen.

(2) Central Leather Research Institute, Madras, India, on the reaction of polyphenolic tanning compounds with hide proteins (collagen).

(b) Chemical modification of hides involves 6.0 p.m.y. at Wyndmoor. This program is concerned with research on the reactivity of hide proteins with various organic and inorganic chemicals in the development of new products intended for specific uses.

(c) New and improved processing involves 8.0 p.m.y. at Wyndmoor. Research is aimed at developing better methods for unhairing animal hides and skins, for imparting deterioration resistance to leathers, and for processing hides into leathers possessing special properties.

Additional research under P.L. 480 grants is in progress at the following foreign institutions:

(1) British Leather Manufacturers Association, Surrey, Great Britain, for a study of deterioration of leather by sweat, chemicals and heat.

(2) Experiment Station for Leather Products, Naples, Italy, for research on the cause of "red heat" and on improved tanning methods for United States hides.

(3) Central Leather Research Institute, Madras, India, on the interrelation of hide quality with the rate of tanning and efficiency of tanning.

(d) Utilization of animal protein residues involves research in cooperation with the National Renderers Association, who support one Senior Fellow and one Junior Fellow at Wyndmoor, Pa., to conduct basic studies on the evaluation of meat and bone meal fractions of residues from fat rendering operations. The Department of Agriculture participates in this program to the extent of supplying supervisory and research leadership amounting to 0.2 p.m.y.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE RESEARCH

A. Chemical and Physical Properties and structure of Hides and Collagen

1. Reversible shrinkage in leather. Studies on the mechanism by which epoxy resins impart the desirable property of reversible shrinkage to leather have been advanced by the use of a very sensitive organic chlorine method capable of detecting minute quantities of the resins in ion exchange column effluents. Conditions have been established for fractionating acid hydrolysates of epoxy resin-treated collagen. A number of fractions containing epoxy resin were collected, but all did not give positive tests for α-amino acids indicating that either the α-amino nitrogen or the carboxyl group took part in the resin-collagen reaction.

Previous investigations showed that the amino acids tyrosine, lysine and hydroxylysine form very stable combinations with the resin. Derivatives of these amino acids and the epoxy resin have been synthesized for use as model systems in probing the complex nature of the mechanism involved in the reversible shrinkage phenomenon.

2. Effect of lipid components on hide properties. The high fat content of domestic cattlehides makes them undesirable for processing into many types of leather. It is conceivable that the lipids are distributed so that certain sections or layers of the hide could be used. Five successive stratigraphic layers of shoulder and butt areas have been analyzed by column chromatography. Phospholipids were predominant in the second and third layers, and wax esters in the top two layers. The data indicate some differences in lipid composition among different sectors of hide. Additional information may suggest tanning and processing modifications to make these hides suitable for specific types of leathers.

3. Noncollagenous proteins of cattlehide. The University of Cincinnati has initiated contract research investigations on the effect of noncollagenous proteins on leather quality and perishability. The extraction of these proteins during the brine-curing step was measured. A brine of 10% salt content extracted most but not all of the protein. Conditions for maximal extraction are being established so that the presence or absence of these proteins can be related to hide quality.

4. Dynamic mechanical tester. The new basic dynamic testing apparatus designed and constructed through contract research at the Franklin Institute has given reliable and reproducible measurements in preliminary tests on rigid plastics, rubbers and leathers. Modifications in the design of the temperature box and cooling system have improved the high frequency performance of the instrument. The addition of automatic data output to simplify use of the equipment was considered but rejected because such automation would decrease accuracy and sensitivity. This new tool promises to be of great value in studying the fundamental physical properties of hides and

fatty-derived polymers and in obtaining new information about the basic molecular principles governing their behavior.

5. Chemical and physical properties of collagen. Solutions of collagen can now be made which are monodisperse and of sufficiently high concentration to yield meaningful data in physical tests. Determinations of sedimentation coefficient, viscosity, optical rotatory dispersion, and electrophoretic mobility have been carried out while varying the parameters of pH, ionic strength, and electrolytic composition of the solution. The data have been correlated with the results of equilibrium-dialysis experiments. These fundamental studies should lead to a better understanding of the properties of reconstituted collagen fibrils and are of interest in the development of new products from cattlehides.

The P.L. 480 grantee at the University of Turku, Finalnd, reported further on the specific properties of collagen and gelatin applicable to the development of expanded uses for hides. A method was developed to correlate the amount of collagen present with the tensile strength and chemical composition of experimental tissues. Certain substances known to increase the fragility of collagen, aminoacetonitrile for example, reduce the tensile strength of the deposited collagen during the life of the animal. Anabolic steroids enhance the strength of the collagenous tissue, but only when the animal is in a state of reduced nutrition. A better understanding of the chemical changes pertinent to the conversion of soluble collagen to insoluble fibers may result in medical utilization of collagens.

Studies on the mode of reaction of polyphenolic compounds with collagen are being supported by a P.L. 480 grant at the Central Leather Research Institute in Madras, India. In tanning experiments with original goran liquor (mangrove extract) on chemically modified collagen, the tan fixed could not be correlated with degree of swelling. Addition of sodium chloride to the tan liquors improved the color, the rate of penetration and shrinkage temperature, but reduced the amount of tan fixed. Penetration was complete in the deaminated sample in 5 days but was not achieved in the methylated sample, regardless of pH. Other tan liquors, such as chestnut, quebracho, myrob and wattle, are being utilized in studies on the rate of tanning as related to shrinkage temperature and yield.

Contract research being conducted at the Midwest Research Institute has revealed some fundamental differences in collagen fibers exposed to enzyme vs. lime-sulfide unhairing. Chemical analyses disclosed that more amide nitrogen was lost during lime-sulfide unhairing. Both treatments resulted in an increase in basic groups, possibly due to the rupture of peptide links. End-group analyses did not provide conclusive evidence for the rupture of specific peptide links. In electron microscope studies, the limed collagen specimens showed better staining properties, possibly because of the presence of more basic groups or because of the removal of a coating material surrounding the fibers. If the latter explanation is correct, a method for removing this component by other than lime treatment would be a valuable development in hide processing.

The microscopic changes induced in the fibrous structure of hides and skins by chemical modification and processing operations are now being studied in a new in-house project. Suitable histological procedures have been selected or developed for tissue studies in which the effects of each processing step will be ascertained. Preparations have also been made to use visual and electron microscopy to elaborate specific structural differences in untreated and treated hide collagen.

B. Chemical Modification

1. Improved water repellency for leather. Efforts to establish the optimum conditions under which leathers accept water repellents have continued. The fat-liquoring formulas have been varied and made more compatible with silicone, an economical waterproofing agent. Alkenyl succinic anhydride (Casyl B18) was proved essential to the fat liquor for good emulsion and exhaustion of the oils. Levels of oil above 5% however, were shown to impair the water resistance of the leather. The use of chrome with glutaraldehyde in the tannage gave leathers with lower water absorption properties than straight glutaraldehyde-tanned leather. A high level of water repellency was achieved with a 2-4% silicone treatment on chrome leather retanned with glutaraldehyde and fat-liquored in water, Casyl B18 and tetrahydrofurfuryl alcohol.

In the large-scale tests, conditions are being established for fat-liquoring full cattlehide sides of chrome grain split in preparation for waterproofing. Tests on flexibility, strength and water resistance are being conducted to evaluate the efficiency of the system.

2. Chemical modification with aldehydes. Most of the fundamental studies have now been completed, and the advantages of glutaraldehyde tanning in imparting many desirable properties to leather have been well demonstrated. Results have been obtained from rate of tanning studies in which grain split cattlehides were tanned with glutaraldehyde alone. Fixation of 3% glutaraldehyde on a moist hide basis was sufficient to make good leather. The rate of tanning and amount of aldehyde fixed increased with the pH and was almost quantitative.

Although the dialdehydes are versatile tanning agents and can be used alone to advantage, they offered greatest promise when combined with chrome. Grain split cattlehides from a glutaraldehyde-chrome study were processed in a tannery into finished shoe upper leather of good quality. Chemical analyses for moisture, fat, ash, Cr_2O_3, and nitrogen, and physical tests for tensile strength and elongation proved the experimental leathers comparable to commercial shoe upper leathers. In addition, they were more mellow and more resistant to degradation by perspiration.

C. New and Improved Processing

1. Enzymatic unhairing of hides and skins. The Tanners Council Research Laboratory completed its contract studies and submitted a final report. Under the terms of this contract, enzyme unhaired hides were run through the

usual tanning process in commercial tanneries to produce sole and shoe upper leathers. The experimental leathers were compared with those made from hides unhaired by the regular lime-sulfide technique. The shoe uppers were deficient with respect to temper, grain texture and veininess; sole leathers were deficient with respect to color uniformity and looseness of grain. Chemical analyses of the two types of leather revealed no significant differences that could be attributed to the unhairing treatments. The contractor advises that only minor variations in the processing techniques may be necessary to produce acceptable commercial leather from enzyme unhaired hides.

The post-tanning operations seem to control many of the characteristics of leather produced from the enzyme unhaired hides. Cooperative studies with several tanneries have shown that softer and mellower leathers can be made from chrome-tanned sides when chrome retannage or heavy vegetable retannage is included in the process. However, these leathers are still considerably firmer than regular leather. Other processing modifications may be suggested from the extensive stratigraphic analyses underway.

2. Improving the deterioration resistance of leather. The P.L. 480 grantee at the British Leather Manufacturers Research Association in Surrey, England, continued to make good progress in elucidating the mechanism of the deterioration of leather by moist heat and perspiration. Studies with dialdehydes indicate that stable cross-links can be introduced into collagen resulting in an increased resistance to deterioration. Experiments with glutaraldehyde labelled with radioactive carbon showed that the disappearance of lysine and hydroxylysine correlates fairly well with the uptake of glutaraldehyde. Since chromium does not bind any of the lysine, these findings suggest a method for the determination of bound glutaraldehyde in chrome-retanned leathers. A completely satisfactory test for durability in wear has not yet been designed, but, in a series of leathers tested for their resistance to the combined action of perspiration and moist heat, the glutaraldehyde tanned leathers were clearly superior to chrome leather.

3. New tanning processes. Work has been intensified on the refinement of the glutaraldehyde process so that the tannage can be applied to a greater variety of leathers. Potential new applications include a simultaneous chrome-glutaraldehyde tannage of bellies for work glove leather and a glutaraldehyde tannage of shearling for washable, perspiration-resistant hospital pads. Several more tanneries are now using glutaraldehyde to upgrade their regular production and to make specialty leathers. Glutaraldehyde is the only tanning agent used by one company in making upper leather for alkali-resistant work shoes.

A new *in situ* tannage of acrolein-formaldehyde polymer and resorcinol used under mildly alkaline conditions yields a novelty leather of commercial quality. Commercial promotion of the tannage does not seem likely because of the cost of these chemicals.

A dialdehyde starch tanning process developed at Lowell Technological Institute under contract research is now ready for evaluation. The complete

sequence of operations required to convert pickled sheepskins into finished garment type leathers has been integrated and successfully tested in trial runs.

The P.L. 480 grantee at the Central Leather Research Institute in Madras, India, has initiated research relating hide quality to rate and efficiency of tanning. Preparations have been made for an investigation of the histological, biochemical, bacteriological, tanning and finishing aspects of different types of leathers. A comparative study of six processes for the production of chrome upper leather from cow hides of average quality has resulted in an acceptable standard process for future studies.

4. Investigations on "red heat." The Stazione Sperimentale per l'Industria delle Pelli e delle Materie Concianti of Naples, Italy, has a P.L. 480 grant to investigate the causes and prevention of the "red heat" damage that occurs on salted American Packer hides received abroad. Microbiologists at the Station have ascribed the defect to chromogenic, halophilic, obligate bacteria. Of 237 microorganisms studied extensively, eleven were designated as specific causative agents. These eleven included two new bacterial species and four new varieties. Histologists proved that "red heat" is restricted to the surface area of the flesh side and does not penetrate to the inner layers or corium. Therefore, although the reddened hides are not as acceptable esthetically, their leather-making qualities are not impaired. To test the effectiveness of four chemical compounds which prevented the occurrence of "red heat" in laboratory trials, Armour and Company has agreed to engage in several salt curing experiments on American Packer hides to be shipped to Italy for further testing.

5. Conversion of U. S. cattlehides into sole leather in Italian tanneries.

In previous studies under a P.L. 480 grant, the Stazione Sperimentale per l'Industria delle Pelli e delle Materie Concianti of Italy established that the high fat content of American Packer hides was responsible for difficulties in processing these hides into sole leather. By thorough fleshing to remove adipose tissue and treating with emulsifying solvents, the grantee reduced the fat content to an acceptable level. Use of a more rapid tanning process based on pretanning with either synthetic tannins or dialdehyde starch yielded leathers very similar to those currently produced by Italian tanneries. The translation of these laboratory experiments into industrial scale operations has been undertaken by several Italian tanners working in close cooperation with the Experiment Station scientists.

D. Utilization of Animal Protein Residues

Investigations supported by the National Renderers Association are concerned with evaluating the quality of meat and bone meal fat rendering residues by characterization of the proteins present. Special fractionation methods have been devised by which meat and bone meals can be separated into bone and meat fractions. The meat fraction is then divided into protein and nonprotein fractions, and the protein fraction subdivided further into salt soluble and

salt insoluble fractions. Analyses of nine commercial meat and bone meals representative of the various types marketed in this country show great variance in nitrogen, ash and lipid contents. The meat fraction obtained by flotation separation contains 13 to 15% nitrogen as compared to 8 to 11% in the complete meal. Although the flotation process can be used to upgrade meat meals by reducing bone content, amino acid analysis is necessary to ascertain whether any of the essential amino acids are lost in the process.

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* Work done under a P.L. 480 foreign research grant.

AREA NO. 6. POTATOES - PROCESSING AND PRODUCTS

Problem. The potato industry, faced with a continuing decline in the consumption of fresh potatoes, is becoming more and more dependent upon the development of new and improved processed products to maintain markets and to avoid recurring economic disasters. Crop perishability, supply fluctuations, and the inelasticity of demand, result in wide swings in price with even slight surpluses. In producing areas having a substantial processing industry, depressive lows are moderated by advance contracting by processors prior to harvest. However, in many important potato growing areas processing has not yet developed, and vulnerability not only still exists, but is exaggerated by the growing competition of processed potato and other competing food products. A continuing improvement in processed potato products is clearly required if processing is to expand fast enough to offset the progressive decline in use of fresh potatoes.

Lack of adequate knowledge concerning the chemical constituents, physical properties, and enzyme systems in potatoes is limiting development of new and improved processed products and processing methods. Basic research on composition is needed to provide fundamental information on which an applied research program can be systematically and effectively built. Recently-developed techniques make it possible to isolate and characterize the constituents responsible for flavor, color, odor, and texture of many processed food products. Application of these techniques to potatoes and potato products should make it possible to improve the quality of present products, both freshly processed and following storage, and provide a basis for technological and engineering studies in new product development.

USDA PROGRAM - EASTERN REGION

The Department has a continuing long-term program of basic and applied chemical and engineering research on studies related to processing. The work of the EURDD, involving the services of chemists, biochemists, food technologists and chemical engineers at Wyndmoor, Pennsylvania, is conducted in cooperation with the Maine Agricultural Experiment Station and several other stations, which supply potatoes of known cultural history. The chemical research program includes: evaluation of the effects of variety, location of production, storage conditions, and tuber solids content on potato composition with particular respect to nitrogenous constituents; principal acids and other factors related to discolorations such as after-cooking discoloration; preliminary studies on the lipids, which are believed to occupy an important role in storage stability of processed potato products, particularly dehydrated foodstuffs. The Eastern Division's engineering and development research program seeks to improve the quality, nutritive value and storage stability of dehydrated potato products and to develop more convenient types of dehydrated products, such as "instantized" pieces that cook quickly. The Eastern Division is now planning the establishment of a Potato Processing Laboratory in the Red River Valley, the purpose of which will be to conduct

investigations relating variety and other raw material characteristics to quality of established forms of processed potatoes. This new Laboratory will be operated jointly by the Red River Valley Potato Growers Association, University of Minnesota, North Dakota State University and the Agricultural Research Service with the Engineer-in-Charge reporting to Wyndmoor.

The Federal (Eastern Division) scientific effort devoted to research in this area totals 14.4 professional man-years. Of this total, research on chemical composition as related to processing characteristics comprises 7.8 p.m.y. During the year a new line of work was initiated in which the potato after-cooking discoloration pigment will be isolated and studied. Research was started on potato lipids. Research on dehydrated potato products constitutes 2.7 p.m.y. Research on new and improved processing technology amounts to 3.9 p.m.y.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition as Related to Processing

1. Nitrogenous constituents. Samples of 5 leading potato varieties (Katahdin, Russet Burbank, Kennebec, Red Pontiac and Cobbler) grown in 6 of the country's principal producing areas (Maine, New York, Pennsylvania, Red River Valley area, Wisconsin and Idaho) in 1961 and 1962 were analyzed. Great differences in composition were found between varieties and within a given variety grown in different locations and in different years. Major changes in the nitrogen pattern during storage appear to occur in asparagine and glutamine. Results of taste panel studies are being statistically analyzed in the attempt to correlate taste with composition.

Potato protein fractions have been separated into at least 12 bands by gel electrophoresis. This definitely indicates that more potato protein fractions exist than just tuberin and tuberinin, commonly reported in the literature.

Work has been continued on the subject of sugar-amino acid compounds formation in potato chip frying. At least 5 compounds of this type have been tentatively identified as being formed during chip-frying.

2. Basic studies on the after-cooking discoloration pigment. A statistical study was made on potato organic acid data obtained under a previous project (E3 6-27). Valid inverse correlations were found between after-cooking discoloration and organic acid content, particularly with citric acid but also with orthophosphoric acid plus oxalic acid. Analytical data indicate that a protein-iron complex may be an important factor in after-cooking discoloration. Addition of ferrous iron to precipitated, denatured protein causes the latter to turn from white to light gray. This color change is irreversible with HCl, but citric acid addition changes the gray back to white. Addition of chlorogenic acid to the protein-iron complex intensifies the light gray to black. Chlorogenic acid added to potato protein in the absence of iron, however, causes no darkening. These observations on induced blackening support

the idea that the combination of ferrous iron, chlorogenic acid, and potato protein is involved in after-cooking discoloration.

3. Basic studies on potato lipids. The first phase of work under this new project has been concerned with obtaining and evaluating new equipment, extraction techniques, and methods of resolving complex mixtures of lipids. Since the unsaturated potato lipids are very susceptible to oxidation during ordinary drying by heat, extraction of fresh tissue has been tried in the attempt to circumvent use of dried potato. This has led, however, to high values due to extraction of nonlipid substance. Experiments are being made in drying potato tissue under very mild conditions, with exclusion of oxygen, to prepare material for organic solvent extraction of lipids. Thin-layer chromatography experiments on crude potato lipid fractions are now in progress.

B. Dehydrated Potato Products

1. "Instantized" pieces. It is believed that a great potential exists for dehydrated potato pieces that will rehydrate more rapidly than the conventional product. Methods have been developed for partially drying the pieces, rendering them porous by quick pressure buildup in a "gun" followed by sudden release to atmospheric pressure, and finally drying the porous structure to low moisture content. Initial drying of the potato dice (nominally 3/8-inch cubes) is carried out at 180° - 200° F. "Explosion-puffing" of potato dice is best carried out at about 35-40% moisture content and 55-65 p.s.i.g. to maximize uniformity of structure and speed of rehydration. "Instantized" dehydrated pieces will reconstitute in less than 5 minutes in boiling water as compared with 20-30 minutes for present commercial dice. Future research is to be directed toward such factors as "instantizing" of larger pieces, increasing product yield, evaluation of a larger pilot plant puffing gun, study of major potato varieties as raw material, and storage characteristics of the products.

C. New and Improved Processing Technology

1. Potato flakes and flakelets storage. Since the FDA would prefer a limit on the concentration of BHA plus BHT antioxidants in flakes lower than that considered by the industry as optimum for best storage stability, it has become advisable to search for other means of imparting storage stability. Flakelets packed in nitrogen without antioxidant, in a 9-month storage test, kept at least as well as the same product packed in air and containing 23 ppm of BHA plus BHT. Since flakelets can be made without antioxidant and packed in nitrogen cheaper than flakes can be made containing antioxidant and packed in air, the former higher-density product is especially well adapted to military, institutional and export use. Tests on the use of tocopherols to protect against oxidation have been discouraging due to the fact that the tocopherols contribute an off-flavor.

2. Technological research in processing. A limited amount of exploratory work has been conducted on chemical and physical properties of potatoes that

influence the color and texture of frozen French fries. A recording attachment has been obtained for the L.E.E.-Kramer shear tester. Attempts are being made to correlate the peaks in the load vs. resistance (to deformation) curve with texture and other characteristics of raw and French fried slices.

PUBLICATIONS AND PATENTS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition and Physical Properties

Bell, Thomas A., Etchells, John L., Williams, Carlos F., and Porter, William L. 1962. Inhibition of pectinase and cellulase by certain plants. Botanical Gazette, 123, pp. 220-223.

Clayton, Morris J., Thompson, John F. and Zacharius, Robert M. 1963. The identification of γ-L-glutamyl-L-leucine and γ-L-glutamyl-L-methionine in kidney bean seeds (*Phaseolus vulgaris*). Journal of Biological Chemistry, 238, pp. 650-652.

Porter, William L. and Schwartz, Joseph H. 1962. Isolation and description of the pectinase-inhibiting tannins of grape leaves. Journal of Food Science, 27, pp. 416-418.

New and Improved Processing Technology

Eisenhardt, N. H., Cording, J., Jr., Eskew, R. K., and Sullivan, J. F. 1962. Quick-cooking dehydrated vegetable pieces. I. Properties of potato and carrot products. Food Technology, 5, pp. 143-146.

Cording, James, Jr., and Eskew, Roderick K. June 12, 1962. Process for manufacture of rapidly rehydratable dehydrated fruits and vegetables. U. S. Patent 3,038,813.

AREA NO. 7. VEGETABLES - PROCESSING AND PRODUCTS - EASTERN REGION

Problem. Vegetable growing occupies over 3 million acres, with a yearly farm value of a billion dollars. Classic studies have revealed their gross composition (moisture, protein, carbohydrate, fat, minerals) but very little is known of the individual constituents that make up these broad classes, and still less about components outside these classes. This ignorance is a limiting factor in the development of new and improved processing methods and processed products. Technological advances have been hampered by insufficient knowledge of the constituents responsible for the color, flavor, and texture of vegetables and the changes these constituents undergo during processing, storage, and distribution. There is need for basic compositional research to provide the fundamental information on which an applied research program can be developed logically and efficiently. Recently-developed equipment and techniques have made it possible to isolate and characterize constituents that could not have been studied effectively with older procedures. There is also need for application of these results to developmental research on new products and new and improved processing technology.

USDA PROGRAM

The Department has a continuing long-term program employing chemists and chemical engineers in basic and applied research on vegetable processing and products. Research (EU) on inhibitors for pectinolytic and cellulolytic enzymes, extractable from plant sources such as grape leaves was completed at Wyndmoor, Pennsylvania, during the past year. Pilot plant research on new dehydrated vegetable products is carried out at Wyndmoor. Research on new processing procedures to produce better quality canned vegetables which was conducted under contract at the New Jersey Agricultural Experiment Station, New Brunswick, has been completed. The Federal (EU) scientific effort assigned to this area totals 2.2 professional man-years and is currently engaged in research on new and improved dehydrated products and processing technology.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Enzyme inhibitors from plant sources. It has long been known that an inhibitor is present in grape leaves that prevents the enzymic softening of cucumbers during brining. It was found that a tannin of the condensed type is responsible for inhibiting this enzymic system. Work at EU under this project was officially terminated on October 12, 1962. Further research on application of the findings to the pickle industry will be conducted by the Southern Utilization Research and Development Division.

B. New and Improved Dehydrated Products

1. Quick-cooking dehydrated vegetable pieces. Methods have been developed for imparting a porous structure to partially-dehydrated vegetable pieces which speeds up their final dehydration and makes them much more readily rehydrated. Following the first stage of drying, the pieces are heated in a "puffing" gun until the water contained within the piece is superheated with respect to steam at atmospheric pressure. The porous structure is the result of the flashing of water vapor from all parts of the piece. Optimum conditions of moisture in the partially dehydrated piece and optimum pressure in the puffing gun vary somewhat from vegetable to vegetable. Thus for developing uniform porosity and ease of rehydration, 3/8-inch carrot dice should be dried to 30-42% moisture content before placing in the puffing vessel and quickly released to atmospheric pressure when 35-40 p.s.i.g. has been reached.

Sweetpotato dice (3/8-inch cubes) were prepared from Maryland Golden (North Carolina production) and Unit #1 Puerto Rico (New Jersey production) varieties by the explosive-puffing process. These products rehydrated in 2-3 minutes' simmering in water, as compared to 40 minutes for conventionally dehydrated dice. The drying time for the explosively-puffed sweetpotato dice was only 5 hours as against 21 hours for pieces of the same size not subjected to puffing. The explosive-puffing process has also been successfully applied to beets and rutabagas, and preliminary experiments have been made with peas and sweet corn.

C. New and Improved Processing Technology

1. Equipment development for explosive-puffing. A cereal puffing gun, such as is used in producing puffed wheat and rice, is the only piece of equipment commercially available for pilot plant investigation of explosive puffing of vegetable tissue. Since the puffing of cereals and explosive puffing of vegetable tissue are fundamentally different, it has been necessary to make changes in the conventional cereal puffing gun.

A larger and improved stainless steel puffing gun (2.5 feet long, 12 inches in diameter) is now in use to provide cost and other data on the production of high-quality vegetable dice.

2. High-temperature short-time canning. This contract research at the New Jersey Agricultural Experiment Station, New Brunswick, on HTST sterilization of canned Eastern vegetables was completed on July 19, 1962, with attainment of the objective. It was demonstrated that HTST sterilization of a number of vegetables results in some improvement relative to conventionally-canned vegetables provided the HTST products are stored under refrigeration.

PUBLICATIONS AND PATENTS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition as Related to Processing Characteristics

Fitzpatrick, Thomas J., Talley, Eugene A. and Porter, William L. 1962. Reactions of asparagine in phosphate buffer. Chemistry and Industry, pp. 1983-1984.

Heisler, E. G., Siciliano, James and Treadway, R. H. 1962. Potassium content of juice in relation to blackening tendency of tissue. Food Technology, 16, pp. 120-124.

Zacharius, R. M. and Talley, Eugene A. 1962. Elution behavior of naturally occurring ninhydrin-positive compounds during ion exchange chromatography. Analytical Chemistry, 34, pp. 1551-1556.

Dehydrated Potato Products

Eskew, R. K. and Drazga, F. H. 1962. Potato flakelets--a new dense product from flakes. Food Technology, 16, pp. 99-101.

New and Improved Processing Technology

Drazga, Fred H. and Eskew, Roderick K. 1962. Observations on drum drying mashed potatoes. Food Technology, 16, pp. 103-105.

Ross, L. R. and Treadway, R. H. 1962. A new and rapid method for determining fat in French fried potatoes. American Potato Journal, 39, pp. 207-211. Reprinted as "A rapid method for determining fat in frozen French fried potatoes," Quick Frozen Foods, 25, pp. 107-108.

Treadway, R. H. 1962. The potato starch industry in the U.S.A. Die Stärke, 14, pp. 212-218.

AREA NO. 8. APPLES AND OTHER FRUITS - PROCESSING AND PRODUCTS - EASTERN REGION

Problem. Lack of knowledge of the nature and quantities of the various chemical constituents and enzyme systems present in fresh fruits, and of the changes these undergo during processing, is a limiting factor in research designed to develop new and improved products and processing techniques. Knowledge is required on the composition and physical structure of fruits and fruit products, with emphasis on substances responsible for color and flavor, vitamins, and other constituents important in determining consumer acceptance and nutritive value of the products. Composition should be studied in relation to variety, stage of maturity, and environmental conditions of growth: and to changes occurring between harvesting and processing, during processing, and in storage and distribution. Recently-developed equipment and techniques have made it possible to isolate, separate, and identify constituents that could not have been handled previously. As basic information is developed, new processing techniques will be applied in the improvement of fruit products, and in more efficient utilization of by-products from fruit processing.

USDA PROGRAM

The Department has a continuing long-term program involving chemists, biochemists, and chemical engineers engaged in both basic and applied research related to extending the use of fruits in the food processing industries. In the EU program, apple products research, and investigations on the chemistry and cell structure of cherries are conducted at Wyndmoor, Pa. Development of rapidly-reconstitutable dehydrated fruit pieces is also underway at Wyndmoor. Contract research on peaches is in progress at Rutgers University, New Brunswick, and on apple texture at the Maryland Agricultural Experiment Station, College Park. The Federal (EU) scientific effort devoted to research in this area totals 9.2 professional man-years. Of this total, research on chemical composition and physical properties constitutes 4.5 p.m.y., including 0.4 p.m.y. of contract research on apple texture at the Maryland Station.

Research on new and improved food products amounts to 2.2 p.m.y. Research on new and improved processing technology amounts to 2.5 p.m.y., including 0.4 p.m.y. of contract research on peach processing at Rutgers.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemistry and cell structure of cherries for processing. Replacement of much of the hand-picking of red tart cherries by mechanical harvesting seems to be a certainty in the future. Large-scale trials were held in Michigan in the summer of 1962 in which mechanically harvested and hand-picked cherries were compared as to quality of the product for processing. While bruising encountered during mechanical harvesting of the cherries was more extensive than

encountered in hand-picking, it was not excessive. Machine-harvested cherries were found to be of acceptable quality for processing. Laboratory tests show that cherries will tolerate the unavoidable minimum bruising in mechanical harvesting, but rough handling in the steps beyond this point leads to a processed product substandard in quality and yield. Bruising must be kept at a minimum to reduce incidence of scald; in addition, the water used in the storage and hauling tanks should be no warmer than about 60° F. to preserve quality. It was found that aeration of the water is of value in maintaining quality, when it is necessary to hold unbruised cherries overnight above 60°F.

Technical assistance was supplied to the red tart cherry industry in the past year in modifying processing procedures to make the fruit acceptable for export to Western Europe. Over 2000 tons of frozen cherries were shipped to Europe in this first industrial effort to develop an export market.

2. Factors influencing apple texture. Contract research (Maryland Agricultural experiment Station) on the relationship between the insoluble carbohydrates and the texture of processed apple products was terminated during the period. Firmness of Golden Delicious, York Imperial and Stayman Winesap apples was found to be related to the amounts of alcohol-insoluble solids present at harvest and during storage. While starch was largely responsible for the firmness of immature fruit, this constituent disappeared during ripening and firmness then became dependent on hemicellulose content. Consistency of apple sauce was related to the alcohol-insoluble solids content, particularly the starch fraction.

Contract research (Maryland Agricultural Experiment Station) on the relationship between apple cell wall constituents and the texture of processed products has begun with storage and processing of two commercial varieties. No results are yet available to permit appraisal.

B. New and Improved Food Products

1. Rapidly-reconstitutable dried fruit pieces. Dehydrated fruit pieces having a porous, rapid-reconstitutable structure are produced by heating the partially dried (25% moisture) pieces above 212° F. in a puffing chamber, then suddenly releasing the pressure, and finally drying in a conventional manner to 2% moisture content. "Explosion-puffing" of blueberries is conducted preferably at 20-25% moisture content; berries "puffed" at above 30% moisture tend to collapse afterward. A rapidly-rehydratable blueberry should be welcomed by the trade, for example, in prepared muffin mixes. Blueberries dehydrated following explosion-puffing rehydrate in 1 to 2 minutes in boiling water and can then be stirred into the mixture of muffin ingredients. This type of blueberry product offers convenient dry pack in the baking mix container, rapid preparation for baking, and flavorful berries in discrete form in the baked muffins.

Apple pieces "explosion-puffed" at 20-30% moisture are rigid, retain their porous structure, and rehydrate on 2 minutes' simmering in water. Commercially-dehydrated apple pieces (20-30% moisture content) fortunately are

suitable for explosion-puffing. This is important in that an organization planning to make this new type of rapid-rehydrating, dried apple piece need not install a line for preparing material to be exploded in the puffing guns. Cost studies indicate that the new type of dehydrated fruit and vegetable pieces can be produced at only a slight increase above that for the manufacture of conventional dehydrated products.

2. Improved apple cider. Pasteurized cider darkens on storage, with accompanying formation of hydroxymethylfurfural (HMF) and sedimentation. Model systems have been used in which it was shown that $NaHSO_3$ addition to acidified fructose solution reduced the formation of color and of HMF at pH 3 to 4. Addition of glycine to fructose solution in the pH range of 2 to 6 increased color formation and HMF. At pH 6, color formation was increased and $NaHSO_3$ became less effective as a color stabilizer. Future work will deal with utilization of these findings to improve the quality of cider products.

3. Lye-peeling of apples. It was found that lye-peeling of apples is rapid and effective if the skin is dewaxed by hot ethyl or isopropyl alcohol treatment prior to the lye dip, or if alcohol is added to the lye solution. Lye-peeling of apples to be canned would permit much saving of labor and also provide low peeling loss.

C. New and Improved Processing Technology

1. Processing characteristics of eastern peaches. Work on this contract research (New Jersey Agricultural Experiment Station) began in June 1962 and continued through the peach harvest season in New Jersey. Ninety-five new varieties of peaches were investigated to determine processing characteristics. Samples from 53 of these varieties were canned and frozen. Preliminary evaluations of color, texture and chemical composition indicate that several of these new varieties show promise for processing.

PATENTS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

New and Improved Food Products

Cording, James, Jr., and Eskew, Roderick K. June 12, 1962. Process for manufacture of rapidly rehydratable dehydrated fruits and vegetables. U. S. Patent 3,038,813.

AREA 9. TOBACCO - COMPOSITION AND PROCESSING

Problem. Although neither food nor fiber, tobacco nevertheless is grown on about a million acres, and in seven states provided more farm cash receipts than any other field crop in 1960. The farm value is about $1.3 billion. This crop is unique in that it yields about $3.1 billion in Federal and State taxes. Several serious difficulties plague the industry, among them the lack of genuine scientific knowledge of the composition of tobacco and tobacco smoke which can be used to solve many industrial problems. By knowing the chemical factors in the leaf which result in an acceptable smoke, it would become possible to predict accurately the usefulness of a particular tobacco for smoking purposes, and thus solve a long-standing industrial problem. Methods could also be devised to expedite current time-consuming and erratic methods of fermenting cigar tobacco. Finally, more selective studies on tobacco smoke could be made, including the origin and fate of leaf constituents during burning, the formation of substances having physiological effects, and ways of producing smoke of diverse composition.

USDA PROGRAM

The Department has a continuing program involving chemists and biochemists engaged in basic and applied studies of the chemical composition of tobacco leaf and smoke, directed to better understanding of and improvement in tobacco quality, and to improvement in tobacco processing technology.

The Federal work is conducted at Wyndmoor, Pa., and totals 7.5 professional man-years, 2.0 of which are devoted to study of the composition of tobacco leaf, mainly cigarette tobacco types, and 5.5 to chemical composition of smoke, primarily cigarette smoke. In addition, the Cigar Manufacturer's Association of America supports parallel effort at Wyndmoor equivalent to two professional man-years; this is a study of cigar smoke.

In addition, the Department sponsors research under a P.L. 480 grant to the University of Sao Paulo, Brazil. This is a study of the changes which occur in the composition of tobacco leaves during curing and fermentation.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Composition of Tobacco Leaf

The isolation of unique constituents in either Turkish or flue-cured tobacco would provide a chemical basis for identification of the source of tobacco, but results to date are negative. Neutral constituents of the hexane extract of Turkish tobacco and of comparable fractions of flue-cured tobacco appeared quite similar in respect to column chromatographic behavior and spectrophotometric characteristics of isolated fractions. Neophytadiene levels did not correlate with commercial leaf evaluations of various flue-cured tobaccos

and thus will not provide a chemical basis for predicting leaf usefulness for smoking purposes.

B. Chemical Composition of Smoke

1. Cigarette smoke. Construction of a multiple-capacity smoking apparatus was completed. Gas chromatograms indicate that the steam-volatile neutral components, about 66 different compounds, in smoke from blended and unblended cigarette tobacco, containing flue-cured, burley, Maryland and Turkish tobaccos, differed on a quantitative rather than a qualitative basis. Preliminary results show some correlation between the aroma of flue-cured leaves and the levels of volatile neutral constituents therein but inconsistencies are observed.

2. Cigar smoke. The study on the weakly acidic substances in cigar smoke was completed. The major components of this category are phenol, o-cresol, m-cresol, and/or p-cresol, palmitic acid and 3-pyridinol. The last is a new component not previously found in tobacco or its smoke. Other compounds isolated and identified are 3,4-xylenol, 2,5-xylenol, 3,4-xylenol and 3,5-xylenol(or m- or p-ethylphenol.) On the basis of equivalent weights of tobacco smoked, the phenol content of cigar smoke is appreciably lower than that of unfiltered cigarette smoke.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Composition of Tobacco Leaf

Bilinsky, W. R., and Stedman, R. L. 1962. Composition studies on tobacco. XIII. Neophytadiene levels in various types and grades. Tobacco Science, 6, pp. 160-161.

Stedman, R. L., Bilinsky, W. R., and Swain, A. P. 1962. Total hexane and ethanol extractives of tobacco. U. S. Dept. Agr., Agr. Research Service Circ., ARS-73-38, 12 pp.

Chemical Composition of Smoke

Ogg, C. L., Bates, W. W., Cogbill, E. C., Blackmore, R. H., and Petersen, E. L. 1962. Determination of total particulate matter, nicotine, and water in cigarette smoke. J. Assoc. Offic. Agr. Chemists, 45, p. 540.

Schmeltz, I., and Schlotzhauer, W. S. 1962. Nonvolatile acids in cigar smoke. Tobacco Science, 6, pp. 88-89.

Schmeltz, I., and Stedman, R. L. 1962. Pyridin-3-ol in cigar smoke. Chemistry and Industry, 27, pp. 1244-1245.

General

Ogg, C. L. 1963. Report on microchemical methods. J. Assoc. Offic. Agr. Chemists, 46, p. 24.

Ogg, C. L. 1963. Report on tobacco. J. Assoc. Offic. Agr. Chemists, 46, pp. 10-11.

AREA NO. 10. MAPLE SAP AND SIRUP - PROCESSING AND PRODUCTS

Problem. The extensive unused stands of sugar maple are largely in infertile and hilly areas of marginal value to agriculture, areas commonly devoted to small-scale dairy farming. Under proper circumstances, maple sirup could be a seasonal crop of value equal to or exceeding that of other farm products. While the results of previous research, for example, establishing the importance of sanitary collection of sap, have contributed to "modernization" of the industry, much more information is needed so that all operations for the production of high-quality maple sirup and of other maple products can be conducted in a predictable, efficient manner. Not only can the marginal farms be greatly benefited, but the existing maple industry in 14 states can be put on a higher economic plane and modernized to be made competitive with other crop and livestock farming.

USDA PROGRAM

The Department has a continuing program involving chemists, biochemists and microbiologists. These scientists are engaged in both basic and applied research in investigations concerned with the problems of improving sap handling and processing, producing high-quality maple sirup, and developing new outlets for all maple products while lessening the cost of the product. Most of this work is conducted at Wyndmoor, Pa. Contract research on factors affecting the quality of maple sirup at the Ohio Agricultural Experiment Station, Wooster, Ohio,continues.

The Federal scientific effort devoted to research in this area totals 5.5 professional man-years. Of this number 2.9 are devoted to study of the chemical composition and physical properties of maple sap and sirup, including 0.3 under the Wooster contract; 1.2 to microbiology of maple products; and 1.4 to new and improved food products and processing technology.

In the research work cooperation is maintained with personnel of the Federal Extension Service in maple-producing states and with Cornell University.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Flavor components of maple sirup. Gas liquid chromatograms indicate the presence of at least 18 components in the flavor fraction of maple sirup. Currently identified are dihydroconiferyl alcohol, syringaldehyde, and vanillin.

2. Maple sugar sand. Further analyses of samples of maple sirup and statistical evaluation of data pertaining to collection of maple sap and composition of the resulting sirup confirm previously reported correlations and provide additional information. Summarizing, there is a positive correlation between sugar sand concentration in a sirup and (a) high minimum temperature during

the five months prior to sap flow, (b) a northern exposure, (c) highest elevations, (d) late stages of sap flow, (e) total organic acid content of the sap, (f) malic acid in the sap, and (g) calcium in the sap. A negative correlation was found between concentration of potassium in the sap and amount of sugar sand formed during evaporation to sirup.

B. Microbiology

1. Improved maple products through microbial fermentation. In the controlled fermentation of maple sap to modify the sap so that it produces a sirup of intensified maple flavor, or, with a "buddy" sap, so that it produces a "non-buddy" sirup, a standardized dry inoculum would provide greater control of the fermentation. Preliminary tests on the use of acetone-dried *Pseudomonas geniculata* cells as the inoculum are encouraging.

2. Microbiological analysis of maple sirup. In a collaborative study of a standardized procedure for microbiological analysis of maple sirup, the counts of microorganisms obtained by this procedure in fifteen government, university and industrial laboratories varied widely. The collaborative assay will be repeated.

3. Prevention of microbial growth in the taphole. An analytical method, specific for formaldehyde, was developed to determine residual formaldehyde in maple sirup so that processors could be sure that the content did not exceed the limit of 2 parts per million established by the Food and Drug Administration as stipulated in the regulations permitting use of paraformaldehyde in tapholes to prevent microbial and fungus growth.

C. New and Improved Food Products and Processing Technology.

A new maple product was developed and it is being rapidly adopted by the trade. Sirup is concentrated to about 75% sugar and whipped with 1% commercial monoglycerides to give a creamy product for use as a topping, icing or spread. A particularly important feature is that the product can be prepared from all maple sirups, providing an outlet for darker sirups and those of above-average content of invert sugar.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition and Physical Properties

Davis, D. R., Hacskaylo, J., and Dunn, F. 1962. Sugar sands are an important part of maple research. Ohio Farm and Home Research 47, 2.

Davis, D. R., Gallander, J. F., Hacskaylo, J., and Willits, C. O. 1963. The chemical composition of maple sirup sugar sand. J. Food Sci., 28, 2, pp. 182-190.

Microbiology

Frank, H. A. 1962. Influence of low incubation temperature on classification of *Pseudomonas geniculata*. J. Bacteriol. 84, pp. 68-71.

Wasserman, A. E. 1962. A method of counting organisms in maple sirup. J. Assoc. Offic. Agr. Chemists, 45, pp. 558-61.

Wasserman, A. E. and Willits, C. O. 1963. Maple sirup XXI. The effect of temperature and formaldehyde on the growth of *Pseudomonas geniculata* in maple sirup. J. Food Sci., 28, 2, pp. 145-148.

New and Improved Food Products and Processing Technology

Sipple, L., Willits, C. O., and Finch, F. E., Jr. 1963. Arches and burners for oil-fired maple sap evaporators. U. S. Dept. Agr., Agr. Research Service Circ., ARS-73-40, 14 pp.

Wasserman, A. E., Underwood, J. C., and Willits, C. O. 1962. Fluffed maple products - A new use for maple sirup. U. S. Dept. Agr., Agr. Research Service Circ., ARS-73-39, 6 pp.

Willits, C. O. 1962. Report on maple products. J. Assoc. Offic. Agr. Chemists, 45, pp. 556-557.

Willits, C. O. 1963. Report on maple products. J. Assoc. Offic. Agr. Chemists, 46, p. 35.

General

Willits, C. O. 1962. Maple aids in better land use program. National Maple Sirup Digest, 1, 3, pp. 8 and 10.

AREA NO. 11. HONEY - PROCESSING AND PRODUCTS

Problem. Essential pollination of overfifty crops depends almost completely on the honeybee because of changes in agricultural practices over recent years. The importance of the beekeeper in our economy is thus emphasized. As most of his income results from the sale of honey, the beekeeper not only is subject to uncertainties of crop and weather, but must also contend with disease, losses of crop and bees from insecticides, rising costs of needed equipment and materials, lack of trained help, all compounded with uncertain and depressed markets for honey. Because of the relatively small size of operations and the scattered nature of the industry, the honey producer is out-researched, out-promoted and out-advertised by competing sweetening agents. Improved processing methods and equipment, better control of product quality, outlets for lower-grade honey, stable export markets, increased use of honey, both in food manufacture and the home, and increased industrial use of byproducts are all needed to provide an expanding market and encourage the beekeeping industry.

USDA PROGRAM

The Department has a continuing long term program involving chemists and biochemists engaged in both basic studies and the application of known methods and principles to the solution of honey producers' problems. This work is done at Wyndmoor, Pa.

The Federal scientific effort devoted to research in this area totals 4.5 professional man-years. Of this, 4.0 are devoted to enzymes, and 0.5 to new and improved food products and processing technology.

REPORT OF PROGRESS FOR USDA

A. Enzymes.

European countries are an important export market for honey. Among the criteria for acceptance as table honey in Europe are a minimum level of diastase and a maximum level of hydroxymethylfurfural. While these levels were established to discourage exposure of honey to heat during processing, this laboratory showed that significant changes in level of these factors may occur during prolonged storage. Analytical data from time-temperature storage of honey samples for periods of 12 to 18 months were used to calculate reaction rate constants for the destruction of diastase and invertase and for the formation of hydroxymethylfurfural. A reduction of 10 to 15° F. from commonly used storage temperatures reduced enzyme loss by as much as 80-85%, and cut formation of hydroxymethylfurfural to one-third of the level found for the higher temperature. This information is already being applied by some of the industry.

"Inhibine," the bactericidal principle in honey, was shown to be hydrogen peroxide formed by the action of glucose oxidase. Conserving enzyme activity in processing and storage will enhance the formation of this factor. A partial purification of glucose oxidase was achieved.

B. New and Improved Products and Technology.

A simple and rapid method was developed for true glucose determination. This procedure will enable a honey packer to determine the suitability of a given honey for a liquid or a finely-granulated pack.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Enzymes

White, J. W., Jr., Subers, M. H., and Schepartz, A. I. 1962. The identification of inhibine. Amer. Bee J., 102, 11, pp. 430-431.

General

White, J. W., Jr. 1962. Determination of acidity, nitrogen, and ash in honey. J. Assoc. Offic. Agr. Chemists, 45, pp. 548-551.

White, J. W., Jr., Riethof, M. L., Subers, M. H., and Kushnir, I. 1962. Composition of American honeys. U. S. Dept. of Agr. Technical Bulletin No. 1261.

AREA NO. 12 - REPLACEMENT CROPS - UTILIZATION POTENTIAL - EASTERN REGION

Problem. Farmers could achieve economic use of their land if new and profitable crops were available that would have different end-uses than crops presently grown. For example, it would be advantageous to develop a new oilseed crop yielding unique fatty acids that could find industrial use in applications for which acids from presently available domestic oilseed crops are unsuitable.

To develop a new crop, three basic steps are involved: (1) survey of wild plants, in cooperation with plant scientists, to identify those having both potentially valuable components and promising agronomic potential for use in the U.S.; (2) detailed physical and chemical characterization of components and basic research to obtain clues to likely end-uses; (3) selection of the most promising species, followed by additional utilization research to explore uses and demonstrate industrial potential and by additional agronomic research to establish proper cultural practices and select the best strains and varieties.

Only after these steps have been successfully accomplished can a proposed new crop be offered to agriculture and industry for introduction and development. Obviously, a program of this type is a long-range one. Yet such long-range research is necessary if agriculture and the nation are to benefit from availability of the best practical crop plants.

To achieve this objective, survey and characterization work needs to be greatly increased, since the greater the number of species examined, the greater will be the opportunities for finding plants meeting the criteria of high utilization and agronomic potential. Work of the Department has already revealed several promising sources of new potentially valuable water-soluble gums, pulp fibers, and oils containing unique fatty acids such as hydroxy unsaturated acids, capric acid, epoxy acids, and unusual long-chain fatty acids. In order to demonstrate the potential of these new materials, further work is required on their physical and chemical properties and reactions, on processing to obtain maximum recovery from source plants and on byproducts from processing, such as oilseed meals.

USDA PROGRAM

The Department has a continuing program involving chemists engaged in both basic and applied studies directed to the development of profitable new crops.

At Wyndmoor, Pa., work on new crops totals 7.2 professional man-years. Of this, 3.5 p.m.y. are devoted to study of the oil obtained from the seed of the Indian ironweed (*Vernonia anthelmintica*), in cooperation with the Northern Utilization Research and Development Division, the Crops Research Division and the Western Utilization Research and Development Division. The oil contains

epoxy fatty acids, potentially useful industrial chemicals.

An additional 3.7 p.m.y. are devoted, in cooperation with the Crops Research Division, to the assay of agronomic samples of Dioscorea tubers for their content of steroidal sapogenins, and to some supporting work on the assay of Canaigre tubers for their tannin content.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Utilization of Oilseeds Containing Epoxidized Oils

Whole Vernonia anthelmintica seeds can be stored at least 30 months in air at room temperature without change in composition of the oil. Domestic seed appears more difficult to extract than seed of Indian origin, and there are some differences in the composition of the oils.

The extraction procedure has been improved: the seed is first wet with petroleum naphtha, flaked wet, and rapidly extracted to give an oil with a minimum of free fatty acids.

The increasing interest of industry in the oil and its derivatives enhance the potential of the plant, and larger quantities of the oil, as well as pure trivernoln, 1,3-divernolin and vernolic acid are being prepared for testing by industry for use in plastics and other products. Vernolic acid is epoxyoleic acid, $CH_3(CH_2)_4\underset{O}{CHCH}\ CH_2CH{=}CH(CH_2)_7COOH$.

B. Assay of Dioscorea and Canaigre

An additional 61 samples of Dioscorea spiculaflora, supplied by the Crops Research Division, were analyzed and the results reported to Crops Research for consolidation with the agronomic studies of that Division. Because of its gentrogenin content, this species is now sought in the commercial collection of uncultivated material. Gentrogenin is a good starting point for the synthesis of many drugs.

Since agronomic studies have now been completed, this work will soon be terminated.

The analysis of 137 samples of canaigre tuber supplied by Crops Research Division completed experimental work under this project and the project has been terminated. The collaboration with Crops Research resulted in the development of more than a dozen strains with a tannin content of 40% or better, with several having extract purities over 70. The highest one, a progeny of a genetic cross, was a sample containing 46% tannin with an extract purity of 73. A strain of this content and purity has a considerable potential as a replacement crop.

Selected samples will be preserved in a living germ plasm bank in Tonto National Forest, Arizona, and a few high-tannin, high-yielding varieties were planted in quantity, to permit rapid expansion in case of emergency. It has

been established that canaigre strains will retain their high level of tannin on repeated culture.

PUBLICATIONS AND PATENTS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Utilization of Oilseeds Containing Epoxidized Oils

Krewson, C. F., Ard, J. S., and Riemenschneider, R. W. 1962. *Vernonia anthelmintica* (L.) Willd. Trivernolin, divernolin and vernolic acid from seed oil. J. Am. Oil Chemists Soc., 39, pp. 334-340.

Riser, G. R., Hunter, J. J., Ard, J. S., and Witnauer, L. P. 1962. *Vernonia anthelmintica* Willd. Seed oil and salts of vernolic acid as stabilizers for plasticized poly(vinyl chloride). J. Am. Oil Chemists. Soc., 39, pp. 266-268.

Scott, W. E., Krewson, C. F., and Riemenschneider, R. W. 1962. *Vernonia anthelmintica* (L.) Willd. (+) and (-)*threo*-12,13-dihydroxyoleic acid. Chem. & Ind., Dec. 1, pp. 208-209.

Wall, M. E., Serota, S. and Kenney, H. E. November 6, 1962. 16,24-Cyclo-21-norcholene derivatives, U. S. Patent 3,062,850.

Wall, M. E., Serota, S. and Kenney, H. E. January 29, 1963. 16-24-Cyclo-21-norcholatriene derivatives. U. S. Patent 3,076,002.

Assay of Dioscorea and Canaigre

Wall, M. E., Warnock, B. H., and Willaman, J. J. 1962. Steroidal sapogenins. LXVIII. Their occurrence in agave lecheguilla. Economic Botany, 16, pp. 266-269.

General

Datta, P. R., and Susi, H. 1962. Gas chromatographic separation of oxygen-containing terpene compounds on low temperature columns. Anal. Chem., 34, pp. 1028-1029.

Datta, P. R., Susi, H., Higman, H. C., and Filipic, V. J. 1962. Use of gas chromatography to identify geographical origin of some spices. Food Technology, 16, pp. 116-119.

Filipic, V. J., Connelly, J. A. and Ogg, C. L. 1962. A study of the thermistor method for molecular weight determinations. Proceedings 1961 International Symposium on Microchemical Techniques, pp. 1039-1051, New York, Interscience Publishers.

Ogg, C. L., Kelly, R. B. and Connelly, J. A. 1962. Apparatus for safe, oxygen-filled flask combustion. 1962. Proceedings 1961 International

Symposium on Microchemical Techniques, pp. 427-433, New York, Interscience Publishers.

Krewson, C. F. July 17, 1962. N-(2,3,6-trichlorobenzoyl)-L-leucine and herbicidal use thereof. U. S. Patent 3,044,866.

Line Project Check List -- Reporting Period July 1, 1962 to June 30, 1963

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Summary of Progress	Incl. in Area & Sub-Heading
E6 2	Milk products utilization investigations. Program Leadership	Wyndmoor, Pa. & Washington, D.C.		
E6 2-64 (C)**	Improving the stability of butter flavor	Ames, Iowa	No	
E6 2-68*	The chemistry of bacterial spores	Washington, D.C.	Yes	1,2-H
E6 2-70**	Development of new outlets for butterfat	Washington, D.C.	Yes	1,2-C
E6 2-71**	Processing high nonfat solids-low fat milk	Washington, D.C.	No	
E6 2-74	Chemistry of flavor changes during processing and storage of dry milk	Washington, D.C.	Yes	1,2-E-1
E6 2-75***	The development of a commercially feasible process for preparing readily dispersible dry whole milk of good flavor and adequate shelf life	Wyndmoor, Pa.	Yes	1,2-B-1
E6 2-77	Improvement in the quality of Cheddar cheese	Washington, D.C.	Yes	1,2-D
E6 2-79	Factors affecting flavor score and storage stability of foam-dried whole milk	Washington, D.C.	Yes	1,2-B-2,3
E6 2-81****	Removal of radioactive contamination from milk	Beltsville, Md.	Yes	1,2-G
E6 2-81 (Rev.)*****	Removal of radioactive contamination from milk	Beltsville, Md.	Yes	1,2-G
E6 2-82(C)	The chemical nature of stale flavor in aged sterile milk	Urbana, Illinois	Yes	1,2-E-1
E6 2-83(C)	Chemistry of Cheddar cheese flavor	Columbus, Ohio	Yes	1,2-D
E6 2-84	A laboratory and pilot plant study of the effect of chemical additives on the storage stability of evaporated milk	Washington, D.C.	Yes	1,2-A
E6 2-85****	Interactions of milk proteins in solution	Wyndmoor, Pa.	Yes	1,2-F
E6 2-85 (Rev.)*****	Interactions of milk proteins in solution	Wyndmoor, Pa.	Yes	1,2-F
E6 2-87	Methods for making fat-free and low-fat cheese	Washington, D.C.	Yes	1,2-D
E6 2-88	Physico-chemical studies of factors influencing milk fat/plasma emulsion stability	Washington, D.C.	Yes	1,2-A
E6 2-89(C)	Development of improved techniques for evaluating importance of flavors in new concentrated milk products	Corvallis, Ore.	Yes	1,2-B-3
E6 2-90	Improvement of concentrated whole milk products	Washington, D.C.	Yes	1,2-B-3
E6 2-91	Properties of components of milk related to physical changes during processing and storage	Wyndmoor, Pa.	Yes	1,2-F
E6 2-92	Development of increased food outlets for nonfat milk solids	Washington, D.C.	Yes	1,2-B-5
E6 2-93*****	The development of a commercially feasible process for preparing a beverage quality dry whole milk having adequate shelf life	Wyndmoor, Pa.	Yes	1,2-B-1
E6 2-94 (C)*****	Effects of nonfat dry milk on bread yeast	Madison, Wisc.	No	
E6 2-95 (C)*****	Increased protein stability of evaporated milk	Columbus, Ohio	No	
E6 2-96*****	Improving the flavor stability of anhydrous milk fat	Washington, D.C.	Yes	1,2-C
E6 2-97*****	The chemistry of bacterial spores	Washington, D.C.	Yes	1,2-H
E6 2-98*****	Studies on stale flavor in sterile milk and development of means to prevent its formation	Washington, D.C.	Yes	1,2-E-1
E6 2-99*****	Improved sterile whole milk concentrates: The production of reversible sol-gel transformations in high solids sterile concentrates	Washington, D.C.	Yes	1,2-A
E6 2-100 (C)*****	Removal of radioactive strontium from milk on a commercial scale	Beltsville, Md.	Yes	1,2-G
EU P1	Pioneering Research Laboratory, Allergens in Agricultural Products	Washington, D.C.	Yes	1,2-F
EU P2	Pioneering Research Laboratory, Chemistry of Animal Proteins	Wyndmoor, Pa.	Yes	1,2-J

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Summary of Progress	Incl. in Area & Sub-Heading
UR-A7-(60)-5	Milk coagulating enzymes	India	Yes	1,2-D
UR-A7-(60)-11	Sulfur compounds in relation to flavor and stability of milk	India	No	
UR-A7-(60)-13	Buffalo milk in cheese manufacturing	India	No	
UR-A7-(60)-22	Proteose-peptone fraction of milk	India	No	
UR-E8-(60)-1	Growth-promoting factors for lactic acid bacteria	Finland	Yes	1,2-D
UR-E8-(60)-5	Effect of feed components on milk and milk products	Finland	Yes	1,2-E-2
UR-E9-(60)-46	Non-protein nitrogenous constituents of milk	France	Yes	1,2-A
UR-E9-(60)-47	Proteolytic activity of rennin on casein	France	Yes	1,2-D
UR-E9-(10,60)-80	Structure of nucleic acids	France	Yes	1,2-I
UR-E15-(60)-16	Food products from milk and fruit concentrates	Italy	Yes	1,2-A
UR-E21-(60)-7	Increasing vitamin B in cheese	Poland	Yes	1,2-D
UR-E25-(60)-18	Protein destabilization in frozen milk	Spain	No	
UR-E29-(60)-31	Microorganisms in dairy products	United Kingdom	Yes	1,2-D
UR-E29-(60)-41	Studies on selected enzymes of milk	United Kingdom	Yes	1,2-A
UR-S3-(60)-10	Active site of trypsin	Brazil	No	

* Superseded during report year by E6 2-97.
** Discontinued during report year.
*** Superseded during report year by E6 2-93.
**** Superseded during report year.
***** Initiated during report year.

Line Project Check List -- Reporting Period July 1, 1962 to June 30, 1963

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Summary of Progress	Incl. in Area & Sub-heading
E6 5	Meat Utilization Investigation. Program Leadership.	Wyndmoor,Pa. & Washington,D.C.		
E6 5-9 (C)*	A study of the relationship of dry milk solids used as binders to product quality and processing characteristics in meat food products	East Lansing, Mich.	No	3-A
E6 5-15 (C)*	Tenderness reversion in frozen beef as affected by holding temperatures and freezing techniques	Stillwater, Okla.	No	3-B
E6 5-17**	Studies of chemical and microbiological factors involved in the freezing of meats	Beltsville,Md.	No	3-B
E6 5-17 (Rev.)***	Studies of chemical and microbiological factors involved in the processing and storage of meats	Beltsville, Md.	Yes	3-B
E6 5-18(C)	A study of meat pigments and their relationship to lipid oxidation and stability of frozen, cured, and uncured meats	Tallahassee,Fla.	Yes	3-B,D
E6 5-19**	Identification of substances responsible for flavor and aroma in meat	Beltsville, Md.	Yes	3-B
E6 5-19 (Rev.)***	Identification of substances responsible for flavor and aroma in meat	Beltsville, Md.	Yes	3-B
E6 5-20**	Chemical reactions involved in meat curing	Beltsville, Md.	Yes	3-D
E6 5-20 (Rev.)***	Chemical reactions involved in meat curing	Beltsville, Md.	Yes	3-D
E6 5-21 (C)***	Investigations on selected characteristics of connective tissue and modification to improve meat tenderness	Baton Rouge, La.	No	3-A
E6 5-22**	Improving the quality of meat through studies of microorganisms capable of growth near 0° C. with particular emphasis on their nutrition and enzymatic activity	Beltsville, Md.	Yes	3-C
E6 5-22 (Rev.)***	Improving the quality of meat through studies of microorganisms capable of growth near 0° C. with particular emphasis on their nutrition and enzymatic activity	Beltsville, Md.	Yes	3-C
E6 5-23***	Improving the flavor of cured meats through a study of the interrelationships of temperature, curing substances, microbial metabolism, and microbial mutation rates	Beltsville, Md.	No.	3-B
E6 5-24**	Studies of the physical and chemical characteristics of meat structure to obtain basic information needed in developing improved methods of meat processing. Meat protein composition in relation to tenderness and juiciness	Beltsville, Md.	Yes	3-A
E6 5-24 (Rev.)***	Studies of the physical and chemical characteristics of meat to obtain basic information needed in developing improvedmethods of meat processing. Meat protein composition and distribution in relation to tenderness and juiciness	Beltsville, Md.	Yes	3-A
E6 5-26 (C)***	A comprehensive study of fungi associated with meat processing and flavor development in order to develop new products of distinctive flavor characteristics	Ames, Iowa	No	
E6 5-27***	Improving smoked meat products by identifying the substances present in wood smoke that either directly or through reactions contribute to the flavor and aroma of smoked meats	Beltsville, Md.	No	3-D
E6 5-28***	Development of new or improved meat processing methods and of new meat products	Beltsville, Md.	No	3-D

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
UR-E8-(60)-4	Effect of microorganisms on sausage flavor	Finland		
UR-E15-(60)-13	Studies on beef canning	Italy	Yes	3-D
UR-E29-(60)-15	Enzymes attacking animal connective tissue	United Kingdom	Yes	3-D
UR-E29-(60)-18	Accelerated freeze drying of meat	United Kingdom	Yes	3-D
UR-E29-(60)-24	Biochemical properties of pork muscle	United Kingdom	No	

* Discontinued during report year.
** Superseded during report year.
*** Initiated during report year.

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Summary of Progress	Incl. in Area & Sub-Heading
E6 3	Animal Fats and Oils and Special Products Utilization Investigations. Program Leadership.	Wyndmoor, Pa.		
E6 3-53*	Addition of nucleophiles to fatty derivatives	Wyndmoor, Pa.	Yes	4-D-1
E6 3-55***	Epoxidized and hydroxylated fat derivatives for industrial use	Wyndmoor, Pa.	Yes	4-B-1
E6 3-55 (Rev.)****	Long-chain fat derivatives for polymer modification	Wyndmoor, Pa.	Yes	4-B-1
E6 3-56	Phosphorus, sulfur, nitrogen and oxygen containing fat derivatives	Wyndmoor, Pa.	Yes	4-D-2
E6 3-57**	Autoxidation of fatty substances in emulsions	Wyndmoor, Pa.	No	
E6 3-58***	Soap-detergent combinations based on animal fats	Wyndmoor, Pa.	Yes	4-C-1
E6 3-58 (Rev.)****	Soap-detergent combinations based on animal fats	Wyndmoor, Pa.	Yes	4-C-1
E6 3-59(C)	Structural characteristics of triglycerides	Villanova, Pa.	Yes	4-A-3
E6 3-60(C)	Dielectric properties of fatty peroxides	Philadelphia, Pa.	Yes	4-D-3
E6 3-61(C)	Substituted vinyl monomers and polymers	Tucson, Ariz.	Yes	4-B-1
E6 3-62	Synthetic lubricants from animal fats	Wyndmoor, Pa.	Yes	4-B-2
E6 3-63	Organic-inorganic fat compounds for use in plastics	Wyndmoor, Pa.	Yes	4-B-1
E6 3-64	Structure of components and derivatives of animal fats	Wyndmoor, Pa.	Yes	4-A-3
E6 3-65	Fractionation and analysis of lipids	Wyndmoor, Pa.	Yes	4-A-1
E6 3-66	Polymerizable amides from animal fats	Wyndmoor, Pa.	Yes	4-B-1
E6 3-67(C)	Structural characteristics of organic peroxides	Pittsburgh, Pa.	Yes	4-D-3
E6 3-68****	Autoxidation of fatty materials in emulsion	Wyndmoor, Pa.	Yes	4-A-2
E6 3-69(C)****	Spatial interrelations within triglyceride molecules	Not determined yet	No	
UR-E9-(60)-79	Hydroxylated fatty derivatives	Marseilles, France		
UR-E25-(60)-22	Cocoa butter substitutes from animal fats	Madrid, Spain		

* Discontinued during reporting year.
** Superseded by E6-68
*** Superseded during report year
**** Initiated during report year

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Sub-heading
E6 4	Hides, Skins and Leather Utilization Investigations. Program Leadership.	Wyndmoor, Pa.		
E6 4-25 (C)*	Evaluation of enzymic processes for unhairing hides	Cincinnati, O.	Yes	5-C-1
E6 4-26	Reversible shrinkage in leather	Wyndmoor, Pa.	Yes	5-A-1
E6 4-28 (C)	Conversion of dialdehyde-tanned skins to salable light leather	Lowell, Mass.	Yes	5-C-3
E6 4-29	Improved water repellency for leather	Wyndmoor, Pa.	Yes	5-B-1
E6 4-30*	Modification of hides and skins with dialdehydes	Wyndmoor, Pa.	Yes	5-B-2
E6 4-31 (C)	Dynamic mechanical tester	Philadelphia, Pa	Yes	5-A-4
E6 4-32	Composition and properties of animal protein residues	Wyndmoor, Pa.	Yes	5-D-1
E6 4-33 (C)	Evaluation of collagen fibers from enzyme-unhaired and lime-unhaired hides	Kansas City, Mo.	Yes	5-A-5
E6 4-34	Processes for making commercial quality leather from enzyme-unhaired hides	Wyndmoor, Pa.	Yes	5-C-1
E6 4-35	New tanning processes for producing leathers of superior durability	Wyndmoor, Pa.	Yes	5-C-3
E6 4-36	Effect of electrolytes and lipid components on hide properties	Wyndmoor, Pa.	Yes	5-A-2; 5-A-5
E6 4-37 (C)	Noncollagenous proteins of cattlehides	Cincinnati, O.	Yes	5-A-3
E6 4-38 (C)**	Preparation and properties of dispersed collagen sols	Undetermined	No	
E6 4-39**	Microscopic investigation of skin and leather structure	Wyndmoor, Pa.	Yes	5-A-5
E6 4-40 (C)***	Abnormalities of leather characterized by a depleted, mush texture	Cincinnati, O.	No	
E6 4-41**	Physical properties of collagen and leathers	Wyndmoor, Pa.	No	
E6 4-42**	Addition of new reactive sites to hide proteins	Wyndmoor, Pa.	No	
UR-A7-(60)-17	Reaction of polyphenolic tanning compounds with hide proteins (collagen)	Madras, India	Yes	5-A-5
UR-A7-(60)-18	Interrelation of hide quality with rate of tanning and efficiency of tanning	Madras, India	Yes	5-C-3
UR-E8-(60)-3	Fractionation of gelatin and collagen	Turku, Finland	Yes	5-A-5
UR-E15-(60)-5	Microbial damage to exported U. S. hides	Naples, Italy	Yes	5-C-4
UR-E15-(60)-7	Tanning studies on American hides	Naples, Italy	Yes	5-C-5
UR-E29-(60)-2	Deterioration of leather by sweat and heat	Surrey, England	Yes	5-C-2

* Discontinued during report period.
** Initiated during report period.
*** Contract negotiated, but not signed as of June 30, 1963.

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Summary of Progress	Incl. in Area & Sub-heading
E3 6	Potato and Other Vegetable Utilizations - Eastern Region. Program Leadership.	Wyndmoor, Pa.		
E3 6-36 (C)*	Evaluation of the quality improvement in Eastern canned vegetables through use of high-temperature short-time processing	New Brunswick,N.J.	Yes	7-C-2
E3 6-38*	Studies on plants containing inhibitors for pectinolytic and cellulolytic enzymes	Wyndmoor, Pa.	Yes	7-A-1
E3 6-41	Development of new types of dehydrated vegetables through modification of internal structure	Wyndmoor, Pa.	Yes	7-B-1; 7-C-1

* Discontinued during report year.

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E3 6	Potato and Other Vegetable Utilizations - Eastern Region. Program Leadership.	Wyndmoor, Pa.		
E3 6-37	Development of "instantized" dehydrated potato pieces	Wyndmoor, Pa.	Yes	6-B-1
E3 6-42	Nitrogenous constituents as quality factors in potato processing	Wyndmoor, Pa.	Yes	6-A-1
E3 6-43	Improvement in storage stability of potato flakes and flakelets	Wyndmoor, Pa.	Yes	6-C-1
E3 6-44	Basic composition studies on the lipid fraction of potatoes	Wyndmoor, Pa.	Yes	6-A-3
E3 6-45	Basic studies on the formation and identity of the after-cooking discoloration pigment	Wyndmoor, Pa.	Yes	6-A-2
E3 6-46 (Pending)	Color and texture of frozen French fried potatoes	Wyndmoor, Pa.	Yes	6-C-2

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E3 3	Apples and Other Fruit Utilization Investigations - Eastern Region. Program Leadership.	Wyndmoor, Pa.		
E3 3-27 (C)*	Utilizing fruit essences and concentrates in frozen desserts	College Park,Md.	No	
E3 3-29 (C)*	Relation of pectin, cellulose and other carbohydrates to texture of processed apple products	College Park,Md.	Yes	8-A-2
E3 3-30	Development of improved apple cider	Wyndmoor, Pa.	Yes	8-B-2
E3 3-32	Rapidly-reconstitutable dried fruit products	Wyndmoor, Pa.	Yes	8-B-1
E3 3-33 (C)	Relation of physical and chemical properties to processing characteristics of eastern peaches	New Brunswick,N.J.	Yes	8-C-1
E3 3-34	Improvement of processed cherries through studies on composition and post-harvest treatments	Wyndmoor,Pa.	Yes	8-A-1
E3 3-35 (C)	Relation of apple cell wall constituents to textural quality of processed products	College Park,Md.	Yes	8-A-2

* Discontinued during report year.

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Summary of Progress	Incl. in Area & Sub-heading
E5 3	Tobacco Investigations. Program Leadership.	Wyndmoor, Pa.		
E5 3-5	Acids and bases in cigar smoke	Wyndmoor, Pa.	Yes	9-B
E5 3-6	Composition of cigarette smoke	Wyndmoor, Pa.	Yes	9-B
E5 3-7	Composition of oxidation products and related substances of tobacco	Wyndmoor, Pa.	Yes	9-A
UR-S3-(50)-4	Fermentation studies on tobacco	Brazil	No	

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E5 1	Sugars and Sirups Investigations. Program Leadership	Wyndmoor, Pa.		
E5 1-60*	Microbial fermentation of sap and selective screening for organisms that impart desirable characteristics	Wyndmoor, Pa.	Yes	10-B-1
E5 1-67(C)	Improvement in processing and quality by study of factors affecting sugar sand formation	Wooster, Ohio	Yes	10-A-2
E5 1-74	Chemical compounds responsible for maple flavors; precursors of maple flavor	Wyndmoor, Pa.	Yes	10-A-1
E5 1-76**	Improvement of the quality of maple products through a study of the fermentation-induced biochemical reactions involved in the formation of maple color and flavor	Wyndmoor, Pa.	No	

*Discontinued during report year.
**Initiated during report year.

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E5 1	Sugar and Sirups Investigations. Program Leadership.	Wyndmoor, Pa.		
E5 1-62(C)*	Investigation of the biological activities of honey	Tucson, Ariz.	No	
E5 1-68	Isolation, characterization and properties of honey enzymes	Wyndmoor, Pa.	Yes	11-A

*Discontinued during report year.

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E5 5	New and Replacement Crops Utilization Investigations. Program Leadership.	Wyndmoor, Pa.		
E5 5-31	Development of plant sources of precursors for steroid hormones: assay of agronomic samples for steroidal sapogenins	Wyndmoor, Pa.	Yes	12-B
E5 5-39	New crop seed epoxy-containing oils	Wyndmoor, Pa.	Yes	12-A
E5 5-43*	Development of canaigre as a source of tannin	Wyndmoor, Pa.	Yes	12-B

* Discontinued during report year.